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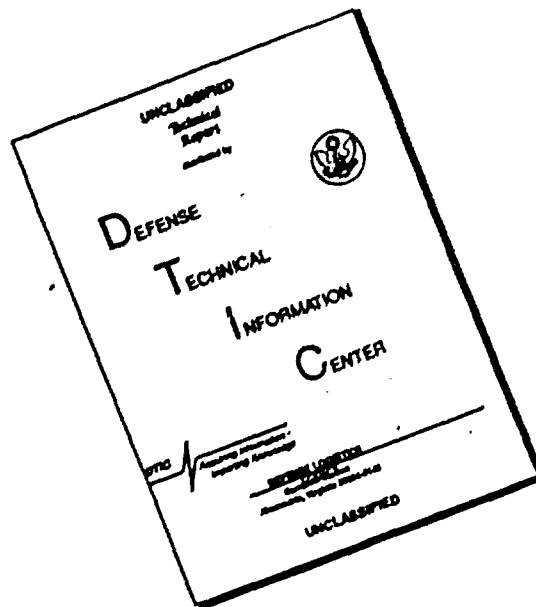
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A DIVISION OF NORTH AMERICAN AVIATION, INC.

CANOGA PARK, CALIFORNIA

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Volume III

31

ADVANCED TECHNOLOGY FOR
PROPULSION SYSTEMS,
FINAL REPORT,
REFRACTORY MATERIALS

ROCKETDYNE

A DIVISION OF NORTH AMERICAN AVIATION, INC.

6633 CANOGA AVENUE
CANOGA PARK, CALIFORNIA

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FOREWORD

This report is the third volume of a four-volume final report submitted in accordance with technical directions set forth by the Bureau of Naval Weapons under Letter Contract NOW 60-0445-c.

ABSTRACT

This report encompasses a general discussion of refractory materials resulting from an investigation performed to disclose the current materials and their properties available to support design and analysis of advanced rocket motor components. The absence of numerous material properties emphasizes the necessity of intensive material research before these materials can be applied to particular designs efficiently.

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INTRODUCTION

A comprehensive investigation of various refractory materials has been performed in an attempt to find the most desirable materials applicable to solid rocket motors. Since the requirements for materials have become so broad in the solid propulsion industry, it is now essential to consider nonmetallic as well as metallic materials. These materials must exhibit high melting points, good thermal conductivity, capacity to absorb heat, erosion resistance, and relative chemical immunity to combustion products.

Problems of coating refractory metals in solid rocket motors to prevent oxidation do not occur because most current rocket exhausts consist of H_2O , CO , CO_2 , and Al_2O_3 which result in a reducing atmosphere. This advantage, however, is absorbed by the disadvantage of the high temperatures encountered with high-performance aluminized propellants being very close to or over the melting point of most refractory materials. The gas also exhausts at very high velocities, being both corrosive and erosive.

In order to withstand the extremely high temperature and erosion conditions encountered while using high-performance aluminized propellants, many new materials are being developed. The more established refractory metals and many carbides, nitrides, borides, oxides, silicides, sulfides, intermetallic compounds, and different types of graphites are being investigated by various companies to establish the physical and mechanical properties which may allow them to be applicable in the propulsion industry. In addition, some cermets, cemented carbides, and cemented oxides are being developed.

Physical and mechanical room-temperature properties for most of the refractory materials are tabulated in this report, and where discrepancies from various sources occurred, the most acceptable value (marked with an asterisk) was recorded. In addition to the properties, a brief description of the various materials from the limited sources of the writer have been introduced. Although reasonably complete information is available for graphite and the refractory metals, relatively little is known about the properties of the other refractory materials since only recently has the need arisen.

It should be pointed out that the physical and mechanical properties found in Tables 1 through 18 have in many cases been measured from controlled samples produced by various methods. As a result, the values (for the various properties) disclosed in this report are reasonable but should be used as preliminary data only. When a critical design analysis becomes necessary, a thorough study should be made to obtain the properties of the (commercially produced) material in question.

The greatest variance in properties occurs with the carbides and some other compounds which are formed by powder methods. The commercially produced materials are often the result of arbitrary mixtures of particles generally of unknown size distribution and containing small percentages of unknown chemical content which may consist of any number of impurities or unidentified phases. Also, the sintering or hot-pressing is generally performed under protective atmospheres which may contain an unknown amount of active gases. The combination of these small variances and some other variances such as peculiar additives used as intermediate binders introduced by some material producers will vary the established material properties to the extent that they may not perform satisfactorily.

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High-temperature physical and mechanical properties are presented for some of the refractory materials as determined by availability and limited experimentation in some areas: this data is presented in Fig. 1 through 28. The data presented is intended to represent the properties of the subject materials more as a trend rather than concise design values. The tables and illustrations are presented in consecutive order at the end of this report.

Material suppliers and fabricators, and methods of fabrication for some of the materials currently used in rocket nozzles are briefly discussed.

REFRACTORY MATERIALS

REFRACTORY METALS

TUNGSTEN (W) OR WOLFRAMIUM

Tungsten metal belongs to the chromium group and is steel-gray in color. Tungsten has particular promise for high-temperature applications because it has the highest melting point of all metals (3410 C, 6170 F) and exhibits exceptional strength at high temperatures. It has the highest recrystallization temperature of all metals, recrystallizing at 2700 F. At room temperature, recrystallized tungsten has an ultimate tensile strength of 90,000 psi. The tensile strength for tungsten ranges from 70,000 to 500,000 psi, depending on the particular cold-worked state of the material. The ductile-brittle transition zone for tungsten ranges from 600 to 650 F. Except for fine wire, tungsten must be hot-worked. At room temperature, recrystallized tungsten exhibits zero ductility. The annealing temperature is 1830 F in a protective atmosphere.

While it is possible to machine, mill, and bore tungsten, better results are obtained by using grinding wheels to remove or cut the material. Punching and shearing must be carried out hot, requiring sharp tools to prevent edge fracture. Tungsten cannot be cast by conventional methods because of its high melting point, and billets cannot be formed by any conventional method until its crystalline structure, as cast, has been broken down by extrusion.

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Tungsten resists most acids and alkalis to 212 F, attacked by nitric-hydrofluoric mixture at room temperature and by aqua regia at 212 F.

This metal-like molybdenum oxidizes catastrophically; however, the oxide it forms is effectively nonvolatile up to about 1830 F. Tungsten is available in bar, rod, wire, sheet, powder, and fabricated forms. The pure metal costs approximately \$8.00 per pound, being relatively abundant. Billets cost \$25 per pound.

Various discrepant values for the coefficient of thermal conductivity and the boiling point have been reported as follows:

Coefficient of Thermal Conductivity

Reference 12 reports	96.6	Btu/hr ft F
Reference 14 reports	75.	Btu/hr ft F
Reference 1 reports	96.	Btu/hr ft F
Reference 13 reports	103.7	Btu/hr ft F

Boiling Point	Reference 12 reports	6700 C
	Reference 22 reports	5900 C

RHENIUM (Re)

Rhenium metal is a member of the manganese family of metals, and is silvery-white in color. Rhenium is a very hard metal but is still ductile and malleable. The transition temperature is believed to be below room temperature. It does not become brittle after prolonged high heating like tungsten; its hardness decreases only slightly from room temperature to 1470 F. Rapid recrystallization initially appears to take place at approximately 1830 F, and above 2730 F the effect of cold-working is nil.

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Rhenium has a hexagonal close-packed crystalline structure, giving it superior strength-retention on a homogeneous temperature basis than those of non-close-packed metals. Unfortunately, for metals melting above 2000 C (3630 F), only rhenium and the refractory platinum metals possess such structures. The linear thermal expansion coefficient for pure rhenium is 6.6×10^{-6} per C.

As an alloying agent, it adds ductility and increases strength to other refractory metals. It is extremely hard and cannot be machined, even with carbide tools. Because of this characteristic, it may have useful surface-coating applications. It has a high rate of work-hardening. Arc-cast rhenium has been somewhat successfully cold-worked, and sintered rhenium bars have been swaged well.

The metal is stable in air and resistant to many acids, but is soluble in nitric acid. The main limitation of rhenium for high-temperature applications is its extremely poor oxidation resistance. The oxide which forms Re_2O_7 , has a melting point of 570 F which seriously limits the use of rhenium at elevated temperatures except in an inert atmosphere.

Rhenium metal is available in sintered bars, powder, and fabricated forms. The price is high at present, ranging from approximately \$680.00 per pound for powder and \$780.00 per pound for sintered bar to higher prices for fabricated forms. The metal is restricted by a rather limited supply.

TANTALUM (Ta)

Tantalum is a bluish-gray metal with a bright luster. It is malleable and very ductile and does not exhibit a transition temperature from ductile to brittle fracture down to as low as -195 C. The tensile strength ranges

from 40,000 to 180,000 psi depending on the annealed or cold-worked condition. The annealing temperature of tantalum is 1950 F in a vacuum. It is easily cold-worked and may be machined like cold-rolled steel with proper lubricants. It is very ductile and can be rolled down from 0.300 to 0.0015-in. without annealing, or it can be drawn into extremely fine wire. The variation of mechanical properties is very pronounced with the atmosphere in which the metal is subjected.

Tantalum is one of the most acid-resistant of the metals and is classed as a noble metal. At very high temperatures, it absorbs oxygen, hydrogen, or nitrogen and becomes very brittle. It will absorb 740 times its own volume of hydrogen, producing a coarse, brittle substance. However, it oxidizes less readily than Re or W, which are the only two higher melting point metals forming the oxide of Ta_2O_5 , which is effectively nonvolatile below temperatures of about 2500 F.

Tantalum can be welded to itself and other metals by resistance or inert arc-welding by special methods. Tantalum is available in bar, rod, wire, sheet, foil, tubing, powder and fabricated parts. Its cost is approximately \$50.00 to \$60.00 per pound.

OSMIUM (Os)

Osmium is a brilliant metal of the platinum family, bluish in color and resembling zinc in appearance. It has the greatest density of any known substance, harder than glass, and very brittle. The metal is not workable, but may be cast at 5000 F.

Osmium oxidizes rapidly in air at elevated temperatures and loses weight due to the volatilization of the low boiling-point oxide, which is

extremely poisonous. It resists common acids at room temperature, but is attacked by aqua regia which, however, does not dissolve it. Osmium is available in cast or sintered parts. It costs approximately \$500.00 per pound.

MOLYBDENUM (Mo)

Molybdenum is a silvery-white metal of the chromium group and is very hard and tough. The tensile strength for molybdenum ranges from 65,000 to 175,000 psi depending on the annealed or cold-worked condition. Generally, molybdenum is brittle at room temperature, gradually becoming ductile up to 2000 F, and brittle again at 2600 to 3200 F, depending on the recrystallization temperature of the particular piece. It cannot be readily heat-treated. Strength must come from cold-working and annealing cycles which must be carefully scheduled so that the piece ends up with the exact amount of cold work. Thin sheet and wire can be cold-worked; however, heavier material must be hot-worked.

Molybdenum can be machined like cast iron, but tool life is shorter. The metal is being processed on a tonnage basis by arc casting. Thin sheets can be resistance-welded and heavier sections inert-arc-welded with special methods. The metal may be forged, extruded, machined, stretch-formed, and spin-formed.

Some disadvantages of molybdenum include those of high reactivity with gases at high temperatures and room-temperature brittleness unless specially processed. Protection of molybdenum from oxidation is also a

major problem. Molybdenum is moderately resistant to acids and alkalis up to 212 F, attacked by nitric-hydrofluoric mixture at room temperature, and attacked by aqua regia at 212 F.

Molybdenum has been characterized as one of the most promising of refractory metals for resistance to molten bismuth and sodium. Molybdenum is available as powder, arc-melted or electron beam-melted billets, bar, rod, wire, sheet, tubing, or fabricated parts. The metal costs approximately \$6.00 to \$25.00 per pound. Billets cost \$18.00 per pound.

COLUMBIUM (Cb) OR NIOBIUM (Nb)

Columbium is platinum-white in color and has a bright metallic luster. The tensile strength for columbium ranges from 40,000 to 100,000 psi, depending on the annealed or cold-worked condition. The annealing temperature is 1950 F in a vacuum. Columbium is the most workable of the major refractory metals, and most fabrication processes can be carried out in the cold condition and with conventional tools. It is also the most ductile refractory metal. It may be machined like cold-rolled steel, with the proper lubricants. Two fabrication problems are those of high affinity for gases in the hot condition and a tendency to stick to dies and tools. An additional disadvantage of columbium is its low modulus of elasticity.

Columbium is resistant to most acids (except hydrofluoric) and to most liquid metals. It is less resistant to alkalis. It has poor oxidation resistance, and oxygen makes the metal harder and stronger and eventually brittle.

Columbium is weldable to itself and other metals by resistance or inert arc-welding, using special methods. The price for this metal ranges from \$55.00 to \$85.00 per pound. Columbium is available in bar, rod, wire, sheet, foil, tubing, powder, and fabricated forms.

Four typical variations in density for various forms of columbium are as follows: (1) pressed, unsintered 6.1 to 6.3 gm/cm³; (2) sintered 9.2 to 9.4 gm/cm³; (3) drawn wire 10.0 to 10.28 gm/cm³; (4) swaged 9.7 to 10.0 gm/cm³.

Discrepancies in values for the melting and boiling points were as follows:

Melting Point:	Reference 6	4530 F
	Reference 12, 13 and 14	4380 F
	Reference 1	4527 F
Boiling Point:	Reference 15	5960 F
	Reference 1	8900 F
	Reference 22	6710 F

IRIDIUM (Ir)

Iridium metal belongs to the platinum family. It is a hard, brittle, nonductile metal. In appearance, it lies between silver and tin. The metal hardness ranges from Brinell 163-350, depending on condition.

As cast, hardness is around Brinell 163, and annealed material has a hardness of Brinell 172. The casting temperature for Iridium is 4600 F. The metal may be hot-worked between 2200 F to 2700 F.

The metal oxides slowly when heated in air. It is unattacked by common acids, including aqua regia, up to 212 F. Iridium can be brazed and resistance-welded. The metal is available in sheet, wire, rod, and powder, and the cost is very high, approximately \$1,300.00 per pound.

RUTHENIUM (Ru)

Ruthenium is a hard, brittle metal of the platinum family, resembling platinum in luster and appearance. It has a Brinell hardness of around 220 in the annealed state. The casting temperature for Ruthenium is 4700 F. The metal may be hot-worked from 2700 F to 4300 F.

Ruthenium may be brazed and resistance-welded. As an alloying element, it has a powerful hardening effect on platinum. The metal oxidizes when heated in air. It is not attacked by common acids including aqua regia up to 212 F. The metal is available in powder form and costs approximately \$400.00 per pound.

HAFNIUM (Hf)

Hafnium is a metal resembling zirconium and is found in nearly all ores of that metal. The tensile strength ranges from 77,000 to 112,000 psi, depending on the annealed or cold-worked condition. The annealing temperature of hafnium is 1380 F in a vacuum or inert atmosphere. It can be hot-worked at 1550 F, or cold-worked 30 percent between anneals. Machinability properties are similar to those of stainless steel.

Hafnium is resistant to oxidizing acids, but is attacked by hydrofluoric acid. The metal has been produced in sheet and rod form.

Discrepancies in values for the melting point and density are as follows:

Melting Point	Reference 12	3400 F
	Reference 13	3580 F
	Reference 6	3870 F
Density	Reference 12	13.0 gm/cm ³
	Reference 6	13.4 gm/cm ³
	Reference 22	12.1 gm/cm ³

RHODIUM (Rh)

Rhodium is a silvery-white metal of the platinum family. The tensile strength ranges from 73,000 to 300,000 psi, depending on the annealed or cold-worked condition. The metal can be hot-worked between 1900 F and 2000 F. It can be cold-worked to a maximum reduction of 30 to 40 percent between anneals. Rhodium becomes malleable at temperatures above 1470 F. The metal casting temperature is 3700 F. It can be brazed and resistance-welded.

Rhodium oxidizes slowly when heated in air. The metal is resistant to most acids, including aqua regia at room temperature. The metal is available in powder, sheet, and wire form. Rhodium costs approximately \$1,400.00 per pound.

VANADIUM (V)

Vanadium metal is a steel-gray metal, capable of taking a very high polish, and is one of the hardest of all metals. This metal has a high melting point and low density, about midway between titanium and steel, making vanadium desirable for structural use. Also, the thermal conductivity is double that of stainless steel and titanium. The metal and its alloys are easily fabricated, and have excellent retention of hot strength up to 1250 F. The strength at 1000 F is nearly as great as at room temperature. The annealing temperature of vanadium is 1650 F in a vacuum of inert atmosphere, and the forging temperature ranges from 1830 F to 2640 F. Vanadium possesses good cold-working properties, but is moderately difficult to machine. The effect of the interstitial elements of carbon, oxygen, hydrogen, and nitrogen on the mechanical properties of vanadium is quite pronounced. The tensile properties of vanadium for different amounts of interstitial content are summarized in Table 1. The weldability is excellent; the metal may be welded or brazed in a protective atmosphere or vacuum.

Vanadium resists sea water, is not affected by moderate-strength hydrochloric and sulfuric acids, but is dissolved by any strength of nitric acid. Almost catastrophic oxidation occurs above 1250 F, but substantial improvements are certain through alloying. The oxide which forms on vanadium and on ductile vanadium-base alloys has a melting point of about 1250 F, which sets the upper temperature of usefulness in an oxidizing atmosphere.

The metal is available in plate, strip, bar, sheet, and wire forms. Its cost is around \$80.00 per pound. Current estimates indicate that vanadium is as abundant in the Earth's crust as nickel and zinc; it is

dispersed through other ore bodies and this, combined with the high production cost of sufficiently pure metal, currently appears to limit its possible use.

CHROMIUM (Cr)

Chromium is a hard, bright, silvery-white metal, capable of taking a high polish. The ultimate tensile strength of chromium ranges from 15,000 to 110,000 psi, depending on the heat treatment and the amount of work-hardening. At 1600 F, pure chromium ruptures in 1 minute at 20,000 psi. Electrically deposited, chromium has a hardness of 9 on the scale, where the diamond is 10, and the metal is much harder than when prepared by other means. Chromium has a good load-carrying ability at high temperature, and is extending the temperature range of super alloys to 1800 - 2000 F.

Brittleness is the basic problem of chromium-base alloys. The values of the transition temperature vary several hundred degrees due to the variation of impurities, fabrication technique, heat treatment, and other factors, making correlation of this property difficult. Ductility in the high purity material is eliminated by recrystallization.

Chromium has excellent oxidation resistance at very high temperatures. The metal is inert to nitric acid, but dissolves in hydrochloric acid and slowly in sulfuric acid.

Discrepancies in values for the density were as follows:

Reference 14	6.39 gm/cm ³ .
Reference 7	7.2 gm/cm ³
Reference 13	7.2 gm/cm ³

ZIRCONIUM

Zirconium exists in two forms, one of which is crystalline, white, metallic, and takes a high luster like nickel, while the other is a bluish-black amorphous powder. The solid metal is obtained from the powder by pressing and heat-treating compacts in vacuum. The tensile strength of Zirconium metal ranges from 24,000 to 90,000 psi, depending on the annealed or cold-worked condition of the metal. The metal annealing temperature is 1550 F, utilizing air for heavy sections and a vacuum or inert atmosphere for light sections. The workability of the metal is similar to that of titanium, and it may be arc or flash-butt welded under an inert atmosphere.

Zirconium has excellent resistance to hydrochloric acid in all concentrations. It resists dilute sulfuric acid, nitric acid in all concentrations, and alkalis. It is attacked by aqua regia. The crystalline metal is stable in air.

Zirconium is available in sheet, rod, tubing, and various shapes. Zirconium metal is about \$20.00 to \$35.00 per pound, and the metal powder is about \$7.00 per pound.

Discrepancies in values for the melting point and coefficient of thermal expansion were as follows:

Melting Point	Reference 6	3870 F
	Reference 1, 12 and 14	3355 F
Coefficient of Thermal Expansion	Reference 1 and 12	3.1×10^{-6} per F
	Reference 13	5.0×10^{-6} per F

THORIUM (Th)

Thorium is a metal resembling platinum in color, and having a high specific gravity and melting point. It is a very soft, ductile metal and is radioactive. The tensile strength ranges from 34,000 to 49,000 psi, depending on the annealed or cold-worked condition. The annealing temperature for thorium is 1380 F in a vacuum.

Thorium can be readily hot or cold-worked; fabricated by forging, rolling, swaging, extruding, or drawing. The metal can be machined like mild steel with or without cutting fluids. It is very difficult to weld, and brazing leaves brittle joints.

Thorium has very poor resistance to atmosphere, water, and most reagents. Atmospheric protection is necessary for high-temperature working. The metal has been produced in rod, sheet, thin-walled tube, fine wire, and foil. Thorium costs approximately \$180.00 per pound.

PLATINUM (Pt)

Platinum is a tin-white, malleable, ductile metal which is harder than silver. The tensile strength ranges from 17,000 to 45,000 psi, depending on the particular cold-worked state of the material. The annealing temperature ranges between 1475 F to 2200 F. The maximum reduction between anneals is 99 percent. The hot-working temperature is 1475 F to 2300 F. The metal casting temperature is 3300 F.

Platinum may be brazed with fine gold or white platinum solder. It can be hammer-welded at 1800 F and can be resistance or oxyacetylene-welded. The metal does not oxidize when heated in air. It resists reducing or oxidizing acids alone, but is dissolved by aqua regia.

The metal is available in sheet, foil, wire, and tubing, with an approximate cost of \$700.00 per pound.

TITANIUM (Ti)

Titanium is a metal of the tin group which resembles iron. The metal is hard, and so brittle that in the cold it can be powdered in an agate mortar, but at a red heat it can be forged. The tensile strength of titanium ranges from 50,000 to 110,000 psi, depending on the cold-worked state of the material. Higher values approaching 180,000 psi have been used, but are not confirmed to the writer's knowledge. The hot-working temperature range for titanium is 300 F to 800 F, and the forging temperature ranges from 1300 F to 1800 F.

The formability of titanium is very good; sheet can be formed at 78 F, and the metal can be fusion-welded.

Titanium has superior resistance to nitric acid, moist chlorine, chlorine solutions, chlorinated organic compounds, and inorganic chloride solutions. It has excellent resistance to corrosive attack by sea water and most chloride salt solutions, being unchallenged by other structural materials. Titanium is available in sheet, strip, plate, tubing, billets, bar, and wire forms. The metal costs approximately \$6.00 to \$11.00 per pound.

PALLADIUM (Pd)

Palladium is a silvery-white metal, less ductile but harder than platinum. The tensile strength ranges from 30,000 to 47,000 psi, depending on the amount of annealing or cold-working. The annealing temperature

is 1475 F, and the hot-working temperature ranges from 1475 F to 2300 F. The maximum reduction between annealing is 99 percent, and the annealed hardness is Brinell 49. The casting temperature for palladium is 3000 F.

The metal can be brazed with oxyacetylene torch using platinum solders, and it can be resistance-welded. Palladium oxidizes when heated in air, and resists hydrofluoric acetic and phosphoric acids. The metal is attacked by nitric, sulfuric, and hydrochloric acids, bromine and iodine. It is also dissolved in aqua regia. The metal is available as sheet, foil, wire, or tubing. The cost of palladium metal is approximately \$270.00 per pound.

BERYLLIUM (Be) OR GLUCINUM (Gl)

Beryllium is a dark gray, very hard metallic element of the magnesium group, which is lighter than aluminum. Beryllium is brittle at room temperature and can be hot-worked at 750 F to 1800 F. The annealing temperature is 1400 F to 2100 F in a vacuum. Machinability of beryllium is difficult due to low ductility. The metal may be brazed with aluminum alloy or silver alloy rods.

Beryllium resists atmosphere at ambient temperatures; is attacked by oxygen and nitrogen at elevated temperatures. resists sea water, and attack in fresh water varies with the air content. Beryllium is available in sheet, plate, rod, bar, tube, and powder forms. The metal costs from \$47.00 to \$75.00 per pound.

URANIUM (U)

Uranium is a hard but malleable metal of the chromium series. It is a lustrous white metal, capable of taking a high polish, and it is radioactive. The metal can be work-hardened to 200,000 psi, most of the working done in the upper alpha phase. 1100 F. It increases the elastic limit and tensile strength of steels, and is also a more powerful deoxidizer than vanadium.

Uranium can be forged, extruded, rolled, swaged and drawn; heating must be done in a protective atmosphere. The metal may be welded or brazed in a protective atmosphere or vacuum. The metal has very poor resistance to atmosphere, water, and most reagents. Uranium has been produced in plate, rod, tube, wire, and foil forms. The metal costs around \$20.00 per pound.

ALUMINUM (Al)

Aluminum was introduced only for physical and mechanical property reference, and due to the fact that it forms a useful oxide.

REFRACTORY CARBIDES

The properties, structure, and composition of metal carbides differ greatly, however, those of groups 4, 5, and 6 (which include Ti, V, Cr, Zr, Nb, Mo, Hf, Ta and W) are quite similar. They are extremely hard, very brittle, retain their mechanical properties at high temperatures, and have very high melting points. In fact, they possess higher melting temperatures than any other class of materials. Although hardness and high temperature stability are desirable properties, brittleness is detrimental. Various schemes of overcoming the detriment of brittleness are the addition of other material, such as metal binders with the compound; special fabrication techniques; closer quality control during production of the compound; and to accept the brittleness and design around it.

The carbide compounds are by no means new; however, many of their properties are still unknown because until now the applications have not demanded thorough knowledge of their properties. Also, testing techniques have not advanced to the stage of those for conventional materials and, as a result, the minimum values necessary to support reliable design analysis are not yet established.

The published properties of the carbides are quite uncertain because the measured values are strongly dependent on the specific sample preparation method. Some of the things which affect the properties are the nature of the sample, chemical composition, purity, grain size and distribution, density, and porosity. When these conditions can be controlled and repeated, then reliable design data may be produced.

The fabrication of the carbides is extremely difficult due to the hardness and abrasion resistance exhibited by these materials. The various carbides

currently being produced are by powdered methods. The physical and mechanical properties for refractory carbides at room temperatures having melting temperatures greater than 1800 C are tabulated in Tables 4 and 5 .

HAFNIUM CARBIDE (HfC)

Hafnium carbide of the chemical formula HfC, with a theoretical carbon content of 6.30 percent, is a gray metallic powder. Like titanium carbide and zirconium carbide, hafnium carbide absorbs, at high temperatures, an amount of carbon beyond that corresponding to the formula HfC, whereby the melting point of the carbide is considered lower. Of all the materials known, only mixtures of hafnium carbide and tantalum carbide melt at a higher temperature than does pure hafnium carbide. The melting point of hafnium carbide is 7030 F.

Hafnium carbide is presently used for nuclear reactor control rods. The vital properties for this application are its high thermal-neutron-absorption cross section and its very high melting point.

Hafnium carbide is available only in finely powdered form; parts must be produced by compaction and sintering. Up to the present, compacted sintered bodies have demonstrated densities of only 80 percent theoretical and precluded development of any significant strengths. A study of the hot pressing variables for this material, temperature, pressure, and time have resulted in fabrication of simple HfC discs with densities of 98 percent of the theoretical density, as reported in the advanced materials section of missiles design and development. The high cost of hafnium carbide prevented investigation of the practical application of the compound until recently.

TANTALUM CARBIDES (Ta_2C and TaC)

There exists two defined carbides of tantalum, Ta_2C having a theoretical carbon content of 3.21 percent, and TaC having a theoretical carbon content of 6.23 percent. Tantalum carbide is an extremely hard, heavy brownish, crystalline material of high melting point. The micro-hardness of tantalum carbide is 1800 kg/mm^2 .

Tantalum carbide is of practical importance in the production of cemented multi-carbide hard metals. In machining tool materials, tantalum carbide, like titanium carbide, reduces the tendency of welding between steel chips and tool material and thus the so-called cratering, which is due to such welding, and the subsequent separation of the welds. Tantalum carbide is only slightly soluble in acids and it burns in air with a bright flash. It may be fabricated by hot pressing, steel die pressing and sintering.

ZIRCONIUM CARBIDE (ZrC)

Zirconium carbide, of the formula ZrC , with a theoretical carbon content of 11.64 percent, is a gray metallic powder. At temperatures above 4410 F, zirconium carbide dissolves carbon so that the melting point of ZrC is lowered from about 6390 F to 4410 F. Upon occurrence of the ZrC phase, the melting point increases sharply with increasing carbon content. The micro-hardness of zirconium carbide is 2600 kg/mm^2 with a load of 50 gm.

Zirconium carbide powder is pyrophoric, and Gangler (Ref. 6) reports excessive oxidation of pure hot pressed ZrC in air at 1800 F. Halogens and oxidizing agents easily decompose the carbide. Above 2700 F, zirconium carbide reacts with nitrogen and forms zirconium nitride.

Zirconium carbide is suitable as a component of cemented hard metals since it forms solid solutions with a number of other carbides. Zirconium carbide is comparatively inexpensive to produce. It may be fabricated by hot pressing, steel die pressing, and sintering.

COLUMBIUM CARBIDE (CbC) or NIOBIUM CARBIDE (NbC)

Columbium carbide, of the chemical formula CbC, with a theoretical carbon content of 11.45 percent, is a gray-brown metallic powder with a violet shine. It is a high-hardness, high-melting compound having a hardness of 2400 kg/mm².

Columbium carbide, as such, has not found technical application, but since it is able to form hard solid solutions with a number of carbides, it has been proposed for cemented hard-metal compositions, including tungsten-free tool material or may be used as an additive to tungsten carbide cutting tools. Its corrosion resistance, density, and electrical properties recommend it for other applications. Columbium carbide is very stable toward acids, and on heating in air, it burns with a bright flash. It may be fabricated by hot pressing, steel die pressing, and sintering.

TITANIUM CARBIDE (TiC)

Titanium carbide, of the chemical formula TiC, with a theoretical carbon content of 20.05 percent, is a light gray metallic powder. An average micro-hardness of 3200 kg/mm² has been found by Kieffer and Kolbl (Ref. 6) and by Hennuber (Ref. 6) while the 2500 kg/mm² has been given from another source (Ref. 7).

Titanium carbide appears most promising for high-temperature applications because at high temperatures, it remains extremely hard and retains a high compressive strength. Its high melting point, low density, high hardness, and high elastic modulus are advantages which recommend it over most metals and alloys. Its low coefficient of expansion and high thermal conductivity result in excellent thermal shock conditions.

Titanium carbide is chemically very stable and is hardly attacked by hydrochloric or sulfuric acid. It is soluble in mixtures of nitric and hydrofluoric acid and also dissolves in alkaline oxidizing melts. It is attacked by chlorine with the formation of chloride or oxychloride. It has relatively good oxidation resistance and the experiments of Dawihl (Ref. 6) on WC-Co and WC-TiC-Co alloys clearly indicate beneficial effect of titanium carbide contents on the oxidation resistance at elevated temperatures. At elevated temperatures, above 2750 F, nitride formation occurs in atmospheres containing nitrogen.

Bonded and compacted with certain metals and ceramics, titanium carbide seems extremely useful. It may be fabricated by hot pressing, steel die pressing, and sintering.

Discrepancies in the values for the density have been reported as follows:

Reference 4	4.25 gm/cm ³
Reference ARDE	4.93 gm/cm ³
Reference 15	4.70 gm/cm ³

VANADIUM CARBIDE (VC)

Vanadium carbide, of the chemical formula VC, with a theoretical carbon content of 19.08 percent, is a gray metallic powder. According to Friederich

and Sittig (Ref. 6)vanadium carbide scratches corundum. Its micro-hardness of 2800 kg/mm^2 has been obtained with a load of 50 gm.

Vanadium carbide is chemically very stable; in the cold, it is attacked only by HNO_3 ; H_2O , H_2S , and HCl are without effect even at red heat; Cl_2 reacts below 930 F. Vanadium carbide is easily produced and very inexpensive.

SILICON CARBIDE, SiC , (Carborundum)

Silicon Carbide is the only carbide which is considered to have good thermal shock resistance. Its flexural strength is practically unaffected up to temperatures of 1500 C (2730 F) with values as high as 70,000 psi reported. Maximum service temperature in an inert atmosphere is 3200 F and in an oxidizing atmosphere is 3000 F.

ALUMINUM CARBIDE (Al_4C_3)

Aluminum carbide is one of the higher melting carbides but little is known of this particular material. Some physical properties may be found in Table 4 .

TUNGSTEN CARBIDES (W_2C and WC)

The carbide of the formula W_2C has a theoretical carbon content of 3.16 percent, while that of WC has a theoretical carbon content of 6.13 percent. The formula W_2C has a micro-hardness of 3000 kg/mm^2 . It is comparatively resistant to acids; however, it is dissolved by hot HNO_3 and, unlike WC , also by a HNO_3 -HF mixture of 1:4 at room temperature. It reacts at 390 F

with chlorine under formation of WCl_6 and graphite. It is attacked by fluorine at room temperature and in a stream of oxygen, it is oxidized to WO_3 at 930 F.

Tungsten monocarbide, WC, is a gray metallic powder. It is the most important constituent of modern cemented hard metals. It is widely employed in single-carbide materials as well as in combination with TiC, TaC, and other carbides. It is, unlike W_2C , not attacked at room temperature by HNO_3 -HF mixtures of 1:4, and starts to react only at room temperature from 1110 F to 1470 F. Against chlorine, it is stable up to 750 F. With Fluorine, it reacts with formation of a flame at room temperature. Upon heating in air or oxygen, WC is slowly oxidized to WO_3 .

The melting points of W_2C and WC are of limited value only as a result of decomposition. Some of the values for the two melting points are reported as follows:

Source	W_2C , F	WC, F
Ruff and Wunsch	-	4710 - 4890
Andrews and Dushman	5220	5040
Friederich and Sittig	-	5220 (decomposes)
Agte and Alterthum	5180 ± 90	5200 ± 90
Barnes	4950 ± 27	-
Sykes	4890 ± 90	4710 (decomposes)
Brewer and co-workers	4950	4770 (decomposes)

MOLYBDENUM CARBIDE (Mo_2C and MoC)

The carbide of the formula, Mo_2C , with a theoretical carbon content of 5.89 percent, is a metallic powder of dark gray color. Mo_2C is relatively

easy to produce, and moreover, since it is cheaper than tungsten carbide, it is of technical importance for cemented hard carbides. Unfortunately, it is considered softer than WC, but in solid solution with other hard carbides, it furnishes useful materials which have found practical application.

Mo₂C is stable in nonoxidizing acids, but is dissolved in nitric acid and aqua regia. Chlorine reacts with Mo₂C only at elevated temperatures, but fluorine reacts with Mo₂C at room temperatures. Upon heating in air, molybdenum dioxide is formed.

The monocarbide of the formula MoC, with a theoretical carbon content of 11.13 percent is, according to Moissan and Hoffman (Ref. 6), a gray powder of metallic luster. Upon heating in air, it is oxidized under formation of MoO₂ and CO₂. It is not attacked by steam up to 1110 F. It is completely dissolved in concentrated HF, decomposed by cold HNO₃ and boiling concentrated HCl, and resists cold KOH and NaOH solutions. It reacts with emission of light with Cl₂. is attacked by Cl₂ at red heat, reacts with Br₂ at elevated temperature, but is only superficially attacked by I₂. Upon heating in air, it is oxidized under formation of MoO₂ and CO₂.

Discrepancies have been reported for the melting points of both Mo₂C and MoC. According to Takei (Ref. 6), graphite is separated upon melting of Mo₂C, and no definite melting point can be given. The discrepancies are as follows:

Source	Carbide	Melting Point, F
Friederich and Sittig	Mo ₂ C	4190
Agte and Alterthum	Mo ₂ C	4870
Schwarzkopf and Kieffer	Mo ₂ C	4800
Research and Development Handbook	Mo ₂ C	4870
Agte and Alterthum	MoC	4882
Schwarzkopf and Kieffer	MoC	4660
Research and Development Handbook	MoC	4880

THORIUM CARBIDES (ThC and ThC₂)

There exists two defined carbides of thorium: (1) ThC having a theoretical carbon content of 4.92 percent, and (2) ThC₂ having a theoretical carbon content of 9.38 percent.

Kieffer (Ref.6) found that thorium monocarbide, prepared from the oxide and carbon, was not wetted by cobalt and did not form solid solutions with other carbides, and concluded that thorium carbide could not be considered as a constituent of technical hard-metal compositions. Thorium dicarbide (ThC₂), according to Langes, "Handbook of Chemistry," ignites rather than melts prior to the melting point as such.

BORON CARBIDE (B₄C)

The percent of porosity is negligible upon physical properties of the material. Its maximum service temperature is 4100 F in an inert atmosphere and 1000 F in an oxidizing atmosphere. Boron carbide is available in densities from 0.069 to 0.091 lb in.³. Boron carbide is currently being produced on an experimental basis by the deposition of boron carbide from a vapor phase onto a substrate which is maintained at elevated temperatures. The properties are expected to be similar to pyrolytic graphite. See Tables 17 and 18 for physical and mechanical properties, and Fig.23 for high temperature thermal conductivity.

URANIUM CARBIDES (UC, U₂C₃, UC₂)

The existence of three carbides has been established, and the available evidence indicates that no carbides lower than UC or higher than UC₂ exist. There is, however, X-ray evidence for a solubility of carbon in UC₂. Above 1800 C (3270 F), U₂C₃ decomposes to form a solid solution of UC and UC₂.

The monocarbide and dicarbide are completely capable of being mixed at elevated temperatures in spite of the fact that the monocarbide is cubic and the dicarbide tetragonal. This type of solid solubility is, however, not without precedence and is discussed in detail by Mallet and co-workers (Ref. 6).

According to Mallet and co-workers, and sesquicarbide does not react with water at room temperature up to 165 F. These authors describe U_2C_3 as a hard and brittle material, and state that in comparison to the sesquicarbide, the monocarbide is brittle and the dicarbide tough.

BERYLLIUM CARBIDE (Be_2C)

Very little information was found for this carbide. It may be fabricated by hot pressing, steel die pressing and sintering, or by hydrostatic pressing and sintering. The coefficient of thermal expansion (77 to 1472 F) is 5.8×10^{-6} per F. This carbide has a compressive strength of approximately 105,000 psi and a modulus of rupture of 16,000 psi.

The thermal shock resistance for this material may be surmised from the following cycling test: four successful cycles being air quenched in the temperature range of 2000-1470 F.

CHROMIUM CARBIDE (Cr_3C_2)

Chromium carbide (Cr_3C_2) is a gray metallic powder. It has a relatively low melting point compared with the other carbides. The carbide may be formed by standard powder metallurgy techniques or cold pressing and sintering.

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Chromium carbide, compacted and bonded with nickel, has exceptionally high resistance to abrasion, erosion, and corrosion. The oxidation resistance of this material at high temperatures is perhaps best of all the metal carbides. It is also very stable toward acids.

Chromium carbide has been suggested as an additional carbide in cemented hard-metal compositions free from, or low in, tungsten. Because of its high solubility in the binder metal cobalt, it has previously not been considered promising. Recently, however, a nickel-bound (cobalt free) chromium carbide with good resistance to abrasion, erosion, corrosion, and particularly high resistance to oxidation at high temperature has been developed. Also, TiC-base compositions containing chromium carbide have proven very promising in the field of heat resistant materials. Chromium carbide may be fabricated by hot pressing or steel die pressing and sintering.

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REFRACTORY NITRIDES

There are several nitrides of high melting point; however, none of these exceed the melting point of tungsten. Potential applications utilizing nitrides are being considered but to date no concentrated effort has been initiated in this direction. Of the refractory nitrides, commercial products are being produced on a limited basis from AlN, BN, and Si_3N_4 . Some physical and mechanical properties may be found in Tables 6 and 7.

HAFNIUM NITRIDE (HfN)

Hafnium nitride is the highest melting nitride known. It is a yellow-brown color, and melts at 6000 F.

TANTALUM NITRIDE (TaN)

Tantalum nitride of chemical formula TaN has 92.81-percent Ta. In general, it is obtained as a dark-gray powder. Sintered bars are brittle and show a blue-gray fracture. At high temperatures, however, they are soft and pliable; e.g., tantalum nitride wires produced by vapor-phase deposition. During melting, tantalum nitride liberates nitrogen.

BORON NITRIDE (BN)

Boron nitride is a light, fluffy white powder. Its X-ray pattern and crystal structure are almost identical with those of graphite, and it is called white graphite. The boron has three electrons; one less than

carbon, and the nitrogen atom has five outer electrons, one more than carbon. Together, in the nitride, they give a result like combining two carbon atoms. Unlike carbon, it is a nonconductor of electricity.

Boron nitride is attacked by acids to form ammonia and boric acid. The melting point is about 5430 F, but it will react with carbon at approximately 3630 F to form boron carbide. The nitride is a good insulator at high temperatures and it is surprisingly stable toward oxidation. Strongly and continuously heated, it will burn with a green-white flame giving off B_2O_3 smoke.

ZIRCONIUM NITRIDE (ZrN)

Zirconium nitride of the chemical formula ZrN, with 13.3 percent nitride, is a yellow-brown powder. High sintered bars of ZrN are brittle and have a lemon-yellow fracture surface. Zirconium nitride does not decompose on melting. It is insoluble in nitric acid; soluble with difficulty in dilute hydrochloric and sulfuric acids, but it is readily soluble in concentrated sulfuric acid. When heated with soda lime, or when boiled with alkalis, zirconium nitride evolves ammonia.

TITANIUM NITRIDE (TiN)

Titanium nitride, of chemical formula TiN, having 77.4 percent titanium, is a light-brown to bronze-brown powder. High-sintered bars of titanium nitride are brittle and have a bronze-yellow fracture. When melted, titanium nitride evolves nitrogen. Titanium nitride is not attacked by hydrochloric acid, nitric acid, or sulfuric acid, but it is readily soluble in aqua regia. When heated with soda lime, or when boiled with alkalis, it evolves ammonia.

URANIUM NITRIDES (UN , U_2N_3 , and UN_2)

Only the monocarbide, UN , is stable at high temperatures. The melting point of UN is listed as 4800 F (2650 C). The use of UN ceramic as a nuclear fuel has been studied by AEC.

THORIUM NITRIDES

Th_2N_3 is unstable at high temperatures, and heating to 1750 C (3180 F) results in decomposition with the formation of ThN . The melting point of Th_2N_3 determined under helium was 2630 C (4770 F).

Thorium nitride combines readily with oxygen and burns easily in air with a great brilliance. Information on this nitride is very uncertain. Data tabulated for this nitride, listed in Tables 6 and 7, were taken from Ref. 15. The uncertainty arises in the information stated above. It is possible that a nitride identified as Th_3N_4 is identical with the hexagonal phase which, according to Chiotti, has a composition close to Th_2N_3 (Ref. 6).

ALUMINUM NITRIDE (AlN)

Aluminum nitride, AlN , forms hexagonal crystals and depending on the temperature of preparation, can be obtained in amorphous or crystalline forms. However, it has a greater tendency to assume a crystalline form. The chemical properties of AlN are very similar to those of Be_3N_2 .

BERYLLIUM NITRIDE (Be_3N_2)

Beryllium nitride, Be_3N_2 , has a cubic (Mn_2O_3 type) crystalline structure. It may be obtained in amorphous or crystalline forms depending on the temperature of preparation.

Beryllium nitride is stable in air and is decomposed only slowly by boiling water. The nitride is readily oxidized at 1832 F. It has a tendency to dissociate at its melting point, 3990 F.

Dilute acids and concentrated alkali hydroxides decompose Be_3N_2 , and dilute solutions of halogen acids react with Be_3N_2 to form solutions of the metal halides and ammonium halides.

COLUMBIUM NITRIDES (CbN and Cb_2N)

In the system columbium-nitrogen, the existence of the nitrides CbN and Cb_2N is established.

Columbium nitride, of the chemical formula CbN containing 87.0 percent of Cb , is a light-gray powder with a yellowish luster. It is insoluble in hydrochloric acid, nitric acid, and sulfuric acid, even at boiling temperature. When heated in air, columbium nitride oxidizes with the liberation of nitrogen and formation of niobic acid. When heated with soda lime, or boiled with strong alkalis, ammonia is evolved. The formula Cb_2N is also stable to acids. When heated with strong lyes or fused alkalis, however, it evolves pure nitrogen rather than ammonia.

VANADIUM NITRIDE (VN)

Vanadium nitride of the formula VN, in pure form with 21.45 percent nitrogen, is a gray-brown powder with a violet luster. Hahn (Ref. 6) describes specimens high in nitrogen contents as metallic, bronze-colored powder. With lower nitrogen contents, the color changes to steel-gray. Vanadium nitride is insoluble in hydrochloric acid and sulfuric acid, but soluble in nitric acid. Strong alkalis decompose the nitride with evolution of ammonia.

SILICON NITRIDE (Si_3N_4)

Silicon nitride, having a melting point of 3450 F, is becoming very useful as a bonding agent for refractory materials. It eliminates the conventional oxide bonding agents, which are subject to solution by high-temperature salts and destruction of the silicon carbide refractory matrix.

Silicon nitride has found commercial use in the bonding of silicon carbide refractories (Ref.4). The silicon-nitride bond material is superior to the conventional clay-bonded silicon carbide in several ways. It has greater shock resistance, greater load carrying ability at high temperatures, and somewhat superior thermal conductivity. Moreover, it can be produced in intricate shapes to close tolerances. Like BN, silicon nitride is very inert chemically.

CHROMIUM NITRIDES (CrN and Cr_2N)

Chromium nitrides of the chemical formulas Cr_2N , with 88.1 percent chromium content, and CrN , with 78.8 percent chromium content, are both yellow powders which are resistant to acids. It is unstable at high temperatures.

REFRACTORY BORIDES

There are a highly refractory group of borides of which ten are mentioned in this report. Hafnium boride is the highest melting of the entire group, melting at 5880 F. Most of the refractory borides are characterized by extreme hardness and high electrical conductivity at high temperatures. Like most inorganic non-metallic materials, however, borides do not exhibit toughness at room temperatures. The borides are hard and have high strength but this desirable property makes them extremely difficult to fabricate, requiring diamond grinding for finishing operations.

The major advantage of borides is that they form stable compounds to very high temperatures and are inert to the oxides. They should be excellent materials for use in composite structures because they are compatible and may be used in contact with other structural materials to very high temperatures. The property data on borides is very skimpy; however, some physical and mechanical properties may be found in Tables 8 and 9.

HAFNIUM BORIDES (HfB and HfB_2)

Hafnium boride is the highest known melting boride and may find useful application in the missile industry. Some physical and mechanical properties for this material may be found in Tables 8 and 9.

TANTALUM BORIDES (TaB_2 , Ta_3B_4 , TaB)

Tantalum boride of the formula TaB_2 , containing 10.68 percent boron, is a gray metallic powder. When heated in red heat, it oxidizes in

air. The diboride is not attacked by HCl , HNO_3 , and aqua regia; it is slowly decomposed by mixtures of H_2SO_4 and HF , and it is rapidly dissolved by fused alkali hydroxides, carbonates, bisulfates, and peroxides.

ZIRCONIUM BORIDES (ZrB_2 and ZrB_{12})

Three intermediate phases have been established: the simple hexagonal diboride, ZrB_2 ; the face center cubic, ZrB ; and the diaboride, ZrB_{12} . The monoboride ZrB has a cubic NaCl -type structure.

Zirconium boride, ZrB_2 , with 19.2 percent boron, is a microcrystalline, metallic, gray powder. According to Andrieux (Ref. 6), it is little attacked by cold HCl , more rapidly by HNO_3 , and is dissolved by aqua regia. It reacts with hot H_2SO_4 , and is readily attacked by fused alkali hydroxides, carbonates, and bisulfates. It reacts violently with brown lead oxide and sodium peroxide.

Glaser (Ref. 6) measured transverse rupture-strength values increasing with density from 8000 to 25,100 psi for ZrB_2 specimens which had been hot-pressed to densities ranging from 87 to 95.5 percent of the theoretical X-ray density value. Zirconium boride is known to be stable in the presence of tungsten, molybdenum and graphite.

COLUMBIUM BORIDES (CbB_2 , CbB , Cb_3B_4)

Columbium boride CbB_2 , with 18.89 percent boron, is a gray powder. Produced by fused-salt electrolysis, it is obtained in the form of small gray crystals of metallic luster.

The formula CbB_2 is not attacked by HCl , HNO_3 , or aqua regia. It is subject to slow attack by hot H_2SO_4 and HF . Heated to red heat in air, it oxidizes. It is rapidly dissolved by fused alkali hydroxides, carbonates, bisulfates, and sodium peroxide. The diboride decomposes, according to Glaser (Ref. 6), at a temperature higher than 5250 F, and in the system $\text{Cb} - \text{B}$, the only phase to melt undecomposed appears to be CbB .

TITANIUM BORIDE (TiB_2)

Titanium boride of the chemical formula TiB_2 , with 31.12 percent of boron, is a gray metallic powder. Produced by fused salt electrolysis, however, it forms fine lamellar hexagonal crystals of yellowish metallic luster. Titanium diboride will scratch corundum and silicon carbide. The measured micro-hardness of titanium is 3400 Kg/mm^2 .

Titanium diboride is not attacked by HCl or HF . It reacts with hot H_2SO_4 , and is easily dissolved by HNO_3 - H_2O_2 , H_2SO_4 - HNO_3 mixtures. It is decomposed by melting alkali hydroxides, carbonates, and bisulfates. It reacts very violently with brown lead oxide and with sodium peroxide.

Between the range of 2900 F to 3600 F TiB_2 tested by the National Carbon Company is the best material available for structural use, exhibiting the highest tensile strength for any material known to date in this range. TiB_2 is known to be stable in the presence of tungsten, molybdenum and graphite.

TUNGSTEN BORIDES (W_2B , WB , W_2B_5)

Tungsten boride of the chemical formula WB is a gray metallic powder. By fused-salt electrolysis, it is deposited in the form of well-developed

crystals of metallic luster. The solubility of boron in tungsten is very low. Tungsten monoboride, according to Weiss (Ref. 6), is not attacked by HCl, but is dissolved by hot H_2SO_4 and HNO_3 . It is readily dissolved by aqua regia, especially in the presence of HF, and by fused alkali hydroxides. It reacts readily with nitrates.

MOLYBDENUM BORIDES (MoB , MoB_2 , Mo_2B , Mo_2B_5)

Molybdenum boride of the formula Mo_2B , with 5.33-percent boron, is a gray metallic powder. Deposited by fused-salt electrolysis, Mo_2B forms bright, slab-shaped crystals, while the acicular MoB is of a somewhat darker color (Ref. 6).

The formula Mo_2B is not attacked by HCl. It is dissolved by cold HNO_3 , by hot H_2SO_4 , and by fused alkali hydroxides and oxidizing agents. According to Steinitz (Ref. 6), Mo_2B decomposes at 3630 F, and all of the molybdenum borides have poor oxidation resistance.

Recent investigations have confirmed the existence of four molybdenum borides Mo_2B mentioned above: MoB with 10.1-percent boron; MoB_2 with 18.4-percent boron; and Mo_2B_5 with 0.22-percent boron.

The microhardness, using a 100-g load is 1660 Kg/mm^2 for Mo_2B , 1570 Kg/mm^2 for MoB , 1280 Kg/mm^2 for MoB_2 ; and, using a 50-g load, is 2500 Kg/mm^2 for Mo_2B , 2350 Kg/mm^2 for MoB , 1200 (?) Kg/mm^2 for MoB_2 , and 2350 Kg/mm^2 for Mo_2B_5 .

VANADIUM BORIDES (VB_2 and VB)

Vanadium monoboride has, according to Blumenthal (Ref. 6), an orthorhombic structure, and is isomorphous with CbB , TaB , and CrB .

Vanadium diboride has a hexagonal structure and, according to Weiss and Blum (Ref. 6), it is insoluble in HCl, HF, and H_2SO_4 , but easily soluble in HNO_3 . It is decomposed by melting alkali hydroxides, carbonates, nitrides, and bisulfates. It reacts violently with brown lead oxide and sodium peroxide. After heating vanadium diboride specimens to the melting point they are, according to an X-ray diffraction study by Glaser, partly decomposed to the monocarbide.

CHROMIUM BORIDES (CrB_2 , Cr_3B_2 , CrB)

Chromium boride of the chemical formula CrB is a gray metallic powder. The formula Cr_3B_2 , produced by fused-salt electrolysis, also forms gray crystals of metallic luster.

According to Andrieux (Ref. 6), Cr_3B_2 is very stable toward HF, HCl, and H_2SO_4 . It is not attacked by HNO_3 , and is very stable toward solutions of alkalis. It is dissolved by perchloric acid and by fused alkali hydroxides and carbonates. Peroxides attack chromium borides much more slowly than other borides. Chromium borides have been developed for use in jet and rocket engines. Tests have shown that they are suitable for sustained use at high temperatures (2000 F) and are capable of resisting stress in engine operations.

REFRACTORY OXIDES

Refractory oxides are currently the most used class of non-metallic refractory materials and are the most understood. They are outstanding for their chemical stability, especially under oxidizing conditions, but their stability at high temperatures is affected by conditions of atmosphere, pressure, and contact with other materials.

On a strength-to-weight ratio, the fracture strength of refractory oxides compares favorably with that of refractory metals. They are known for their low thermal shock resistance which may be overcome by design, such as limitations or control of shapes to alleviate detrimental thermal gradients. Physical and mechanical properties for various refractory oxides may be found in Tables 10 and 11.

THORIUM OXIDE (ThO_2)

Thorium oxide, or thoria (ThO_2) is chemically inert and highly refractory, having cubic crystals. It is marketed as a fine-grained powder, or in small chips, and used for crucibles. It has the highest melting point of all the oxides. The use of thorium oxide as a refractory is limited because of its cost and its sensitiveness to sudden temperature changes, having poor thermal shock resistance. Also, it is radioactive.

The addition of 1 to 2 percent of ThO_2 to molybdenum significantly increases the strength of molybdenum at temperatures above 3500 F. Thoria is insoluble in water, alkalis, and dilute acids, but is soluble in hot sulfuric acid. Like many of the other oxides, thoria is resistant to acids when it is well calcined. Thoria may be fabricated by pressing, tamping, and slip casting. Crucibles and tubes of high purity thoria are available commercially.

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MAGNESIUM OXIDE (MgO)

Magnesium oxide, or magnesia, MgO , is not troubled with crystalline inversions. It is one of the most abundant of the refractory oxides. It is slightly more costly per unit volume than alumina. It has very high thermal expansion.

Magnesium oxide is easily reduced at high temperatures and volatilizes at temperatures much above 4170 to 4350 F. Its maximum useful temperature in an oxidizing atmosphere is 4350 F while in reducing atmospheres, it is limited to about 3090 F. Its stability is poor in a reducing atmosphere, but it is stable in contact with carbon up to about 3270 F, however it reacts highly with carbon as well as with the carbides of some metals at 3630 F.

Magnesia is slowly soluble in water and mineral acids unless calcined highly, but the high-temperature, high density form has a good resistance to mineral acids, acid gases, and moisture. It also is resistant to reaction with neutral salts at high temperatures. Magnesia has been fabricated by slip casting, extrusion, pressing, and even by fusion. One of the major problems of magnesia is its tendency to hydrate in contact with moisture.

URANIUM OXIDE (UO_2)

Uranium oxide, UO_2 , has a cubic crystal with a fluorite lattice. The dioxide is readily oxidized to U_3O_8 , which at temperatures above 1380 F, is again decomposed to a lower oxide. Uranium dioxide is difficult to reduce, and bodies of uranium oxide can be used in either neutral or reducing atmospheres.

Uranium dioxide is insoluble in water but soluble in acid. It is corrosive to some metals, and it is radioactive, but if proper precautions are observed, it may be fabricated safely into refractory bodies. It is a commercial product controlled by the Atomic Energy Commission.

HAFNIUM OXIDE (HfO_2)

Hafnium oxide, or hafnia, HfO_2 , has attractive properties as a high-temperature refractory. It is very similar to zirconia and would be expected to show the same stability and chemical inertness at high temperatures.

Hafnium oxide exists as a solid solution in zirconium oxide or zircon in the amount of 1 to 7 percent. It probably would be preferred over zirconium oxide as a refractory material, but because of its scarcity and cost of separating from zirconia, its use as a refractory oxide is limited.

ZIRCONIUM OXIDE (Zr_2O)

Zirconium oxide, or zirconia, Zr_2O , with a fusion point of 4850 F, has unusually low thermal conductivity. Like alumina, it is stable in both oxidizing and moderately reducing atmospheres; and like zircon, it is stable in contact with many metals and oxides. It can be used at higher temperatures than either alumina or zircon, but is considerably more expensive.

Zirconia, in the pure state, inverts in crystalline structure, which causes disastrous volume change when it is heated or cooled. It may be stabilized by the addition of small amounts of calcia or magnesia to

form a product with moderately good thermal shock resistance and only a small loss of refractoriness. Zirconia is insoluble in water and is resistant to acid and neutral materials at high temperatures, but is attacked by basic oxides to form zirconates. It is acidic.

The crystal form of baddeleyite (natural zirconium oxide), which is monoclinic, is stable at normal temperatures, and this stability is maintained up to about 1832 F. At higher temperatures, the stable crystalline form is tetragonal. When pure zirconia is heated to temperatures above 1832 F, and recooled, it not only undergoes large volume changes with temperature but may not even return to its original dimensions (Ref. 4). Titanium reacts severely with zirconia, as does basic steel slag. When in contact with carbon at 2200 C, in vacuum, or in hydrogen or nitrogen atmospheres at high temperatures, zirconia decomposes to form the carbide, hydride, or nitride.

CERIUM OXIDE (CeO_2)

Cerium oxide, ceria, or ceric oxide, CeO_2 , is a pale yellow heavy powder. The cerium dioxide crystal is cubic. It is a fairly stable oxide and is of interest as a refractory, but has poor thermal shock resistance.

The calcined oxide is insoluble in hydrochloric or nitric acid, except in the presence of a reducing agent, and is also resistant to alkalis. It is stable in air, but may be reduced to a lower melting form (Ce_2O_3 , melting point 1690 C) if heated in other than an oxidizing atmosphere. The melting point of CeO_2 is 4700 F.

CALCIUM OXIDE (CaO)

Calcium oxide, calcia, or lime, CaO, has a cubic crystal with a sodium chloride structure; an amorphous form which changes to the crystalline form at about 750 to 800 F; and a hexagonal form, particularly as a sublimation product.

Calcium oxides low cost, high refractoriness, and stability in contact with metals make it an attractive material for high temperature use. Calcia reacts readily with water, however, to form the hydroxide, and bodies made of calcia will hydrate in air and decompose in a few days. The anhydrous oxide does not react with cold, dry acid gases, but reacts readily when hot. At high temperatures, it reacts with carbon to form the carbide, and it forms salts with all the acidic refractory oxides. One approach to a solution of the problem of stabilizing calcia is to convert the oxide to highly refractory silicates which are resistant to hydration, but the chemical nature of the product is drastically altered.

Calcia may be fabricated by methods similar to those used in fabricating magnesia, but the conditions for processing calcia are somewhat more critical than those for magnesia. Williams (Ref. 4) describes methods for fabricating calcia by pressing and also gives precautions necessary to prevent hydration of the oxide in both the green and fired conditions. The maximum useful temperature in an oxidizing atmosphere is 4350 F. The stability of calcia in a reducing atmosphere is very poor.

BERYLLIUM OXIDE (BeO)

Beryllia, or bromellite, BeO, has a hexagonal crystal structure and is free of inversions. The oxide is toxic, causing a decrease in interest

for use of this material for commercial refractories. Poisoning from beryllium compounds may be extremely serious and even fatal; therefore, precautions must be taken to prevent absorption of the material into the body, particularly by inhalation of dust or fumes.

Beryllia has a great thermal stability and particularly high resistance to reduction. It is relatively weak at low temperatures, but its mechanical properties remain fairly constant up to about 2910 F, and, at this temperature, it is one of the strongest oxides in compression. Its high thermal conductivity, comparable to that of some metals, coupled with a moderate and uniform thermal expansion, makes it very resistant to thermal shock.

The fabrication of beryllia pure oxide refractories by ramming, casting, pressing, and extrusion is described by Norton (Ref. 4). A refractory grade of BeO, about 99.5 percent pure, is available commercially. The principal impurities may be SiO_2 , CaO, Al_2O_3 , Na_2O , and Fe_2O_3 . Also, beryllia pure oxide refractories, which are essentially nonporous, may be produced by a process of sinter recrystallization.

Beryllia is an excellent refractory for containing molten metals due to its high chemical inertness at elevated temperatures. The maximum useful temperature of beryllia in an oxidizing atmosphere is 4350 F. The compound has an excellent stability in a reducing atmosphere.

STRONTIUM OXIDE (SrO)

Strontium oxide, or strontia, SrO , exists in amorphous and two crystalline forms. The common crystalline form is cubic, but a rhombic modification

has also been prepared. It is not listed as a commercial product, but it may be prepared conveniently from the carbonate.

Strontia is stable at high temperatures, but it reacts readily with moisture at room temperature. Bodies of strontia, like calcia, hydrate in air and decompose to powder. It also reacts with carbon dioxide and sulfur dioxide when heated.

YTTRIUM OXIDE (Y_2O_3)

Yttrium oxide, or yttria, Y_2O_3 , has a cubic crystal. It is fairly stable in air and is not easily reduced. It absorbs carbon dioxide and is readily soluble in acids. Yttria is a commercial product, but only produced in small quantities.

LANTHANUM OXIDE (La_2O_3)

Lanthanum oxide, or lanthana, La_2O_3 , exists as hexagonal or cubic crystals. It is a white powder which is used in gas mantles and for absorbing gases in vacuum tubes. Like calcia, it is unstable in air, but unlike calcia, it is not readily available. It is soluble in acids and readily absorbs water.

ALUMINUM OXIDE (Al_2O_3)

Aluminum oxide is referred to as alumina or corundum, Al_2O_3 . In natural or artificial crystalline form, it is used as an abrasive. Pure alumina is a white amorphous powder, or it may be in nearly colorless crystals.

Aluminum oxide is chemically one of the most stable and mechanically one of the strongest of the refractory oxides, particularly at moderate temperatures.

It is insoluble in water, and, if it has been strongly calcined, it is insoluble in both mineral acids and bases. At temperatures as high as 3090 to 3270 F, it is resistant to all gases except fluorine. Alumina is very stable in the presence of both oxidizing and highly reducing atmospheres and can be used in either type of atmosphere at temperatures up to about 1900 C or 1950 C (3542 F) for short periods, but dissociates slowly in vacuum above 1800 (3272 F) to gaseous Al_2O and O_2 .

Shapes of alumina can be formed by slip casting, extrusion, cold or hot pressing, or injection molding, and are matured by dry pressing in steel molds with organic binders or lubricants.

TITANIUM OXIDE (TiO_2)

Titanium oxide, or titania, TiO_2 , is a good refractory and the finely ground material gives good plasticity without binders. Titanium oxide occurs in four allotropic forms: brookite, rutile, and two forms of anatase.

Titanium dioxide is difficult to mature to a dense, strong shape without at least partial reduction to a lower form of oxide. Its use probably should be limited to oxidizing atmospheres because slightly reduced TiO_2 has a relatively low melting point. The lower oxide, Ti_2O_3 , has a melting point of 3450 F, but an intermediate oxide, $2\text{Ti}_2\text{O}_3 - 3\text{TiO}_2$, has a melting point of 2860 F (Ref. 4). Titanium dioxide is insoluble in water and most acids, but is soluble in alkalis.

Titanium oxide is a commercial product, and a high-purity form is produced for the pigment industry.

BARIUM OXIDE (BaO)

Barium oxide, baria, BaO , is a common commercial product. It is generally considered to be amorphous, but hexagonal and cubic forms are known. Little information on its use as a refractory is known. It is poisonous.

Barium oxide is not stable in air, even after being fired at high temperatures. It reacts rapidly with water, and with sulfur dioxide and carbon dioxide when heated. Barium oxide has a strong tendency to form the peroxide (BaO_2) and is therefore a reducing agent.

CHROMIC OXIDE (Cr_2O_3)

Chromic oxide, Cr_2O_3 , crystal belongs to the trigonal division of the hexagonal system. Chromic oxide is isomorphous with corundum, and it forms solid solutions with alumina which are very refractory, but these combinations have poor thermal shock resistance. Chromic oxide is little used as a pure oxide refractory, but widely used as a refractory when it is in combination with alumina and magnesia.

There is little information about the fabrication or the properties of bodies of pure Cr_2O_3 , but this material may be a satisfactory refractory when not subjected to a reducing atmosphere. Cr_2O_3 is a commercial product.

NICKEL OXIDE (NiO)

Nickel oxide or bunsenite, NiO , has a cubic crystal with a sodium chloride structure. It is a commercial product, but is not attractive as a refractory because in reducing atmospheres it is readily reduced to a metal, and in oxidizing atmospheres it oxidizes to Ni_2O_3 at approximately 750 F and goes back to NiO at approximately 1190 F. A volume change, which accompanies this reaction usually causes damage to bodies of NiO . Nickel oxide is insoluble in water and in alkali hydroxides, but is soluble in most concentrated acids.

TANTALUM OXIDE (Ta_2O_5)

Tantalum oxide, Ta_2O_5 , has crystals which are rhombic needles. It is a fairly stable oxide and is a commercial product.

Tantalum oxide is insoluble in all acids except concentrated hydrofluoric, but is soluble in molten alkali carbonates or by fusion with potassium hydroxide or bisulfate. It is not readily reduced at high temperatures, although at very high temperatures, hydrogen reduces Ta_2O_5 to a lower oxide. It does not decompose below its melting point when heated in air. Tantalum is usually found in combination with niobium, but due to the difficulty of separation, high-purity compounds are very costly.

TIN OXIDE (SnO_2)

Tin oxide, SnO_2 , occurs in three crystalline forms: tetragonal, hexagonal, and rhombic. There is a transformation between 750 and 797 F. It has limited usefulness as a refractory because it volatilizes readily

at temperatures above 2730 F. Bodies of tin dioxide have good thermal-shock resistance, however, and are attractive as refractories at moderate temperatures. Tin oxide is easily reduced and therefore probably can only be used in oxidizing atmospheres, and only at temperatures below 2730 F. Tin oxide sublimates at the boiling point.

The dioxide is insoluble in water and has good chemical resistance. It does not react with most acids, is very resistant to molten glass, and is insoluble in alkali hydroxides but soluble in fused alkalis.

VANADIUM OXIDE (V_2O_3)

Vanadium trioxide, V_2O_3 , has crystals which are hexagonal and has a corundum type of structure. Vanadium trioxide, V_2O_3 , with a melting point of 3590 F is readily oxidized to V_2O_5 which has a melting point of 1235 F.

Vanadium oxide is quite resistant to reduction and might be used as a refractory in neutral or reducing atmospheres. It is insoluble in most acids and alkali hydroxides.

ZINC OXIDE (ZnO)

Zinc oxide, or zincite, ZnO , has a hexagonal crystal. It melts at 3588 F but volatilizes readily at temperatures above 3090 F. It is also readily reduced at high temperatures.

Zinc oxide is not used as a refractory, but is soluble in many fused salts and oxides and is an important constituent in glass and glazes. It is insoluble in water, but is soluble in both acids and bases. Zinc oxide is a very common commercial product.

REFRACTORY SILICIDES

Silicides are not of great interest currently as refractory materials; however, the disilicides exhibit good oxidation resistance at high temperatures and also maintain high tensile strength and modulus of rupture at high temperature when most oxides and metals begin losing this property. A very narrow range of applications have been found for the silicides and material properties are practically non-existent; however, some properties may be found in Tables 12 and 13. At low temperatures, silicides exhibit poor oxidation resistance.

Studies of the mechanism of the reaction of Mo and W disilicides in an oxidizing environment at temperatures up to 1700 C (3092 F) or higher show that the oxidation resistance of these materials results from the formation of a glassy coating of silica over the disilicate shape. These silica coatings are firmly anchored to the disilicide base and they are self-healing so that, when a break occurs from impact or other stress application, the coatings will renew itself during continued exposure to an oxidizing atmosphere at high temperatures.

TANTALUM SILICIDE (TaSi_2)

Tantalum disilicide (TaSi_2) is bluish-gray with a metallic luster. The disilicide is oxidation-resistant in air at elevated temperatures, but oxidizes slowly in a stream of oxygen at red heat.

Except for hydrofluoric acid, mineral acids do not attack TaSi_2 . It is attacked by fluorine and chlorine at elevated temperatures, but is practically resistant to bromine. It is readily decomposed by fused alkali.

COLUMBIUM SILICIDE (CbSi_2)

The chemical characteristics are similar to those of vanadium disilicide. The micro-hardness of columbium disilicide is 1050 kg/mm^2 .

Columbium-silicon alloys with more than 60-atomic-percent columbium ignite strongly when heated at 2250 F, and the oxide flakes off. The columbium silicides are, therefore, poorly resistant to oxidation (Ref. 19).

TUNGSTEN SILICIDE (WSi_2)

The disilicide, WSi_2 , prepared by aluminothermic reaction, crystallizes in the form of hexahedral prisms of bluish-gray color and metallic luster. Tungsten Silicide has a measured microhardness of 1090 kg/mm^2 .

Tungsten disilicide is oxidation-resistant in air at elevated temperatures, but oxidizes slowly in a stream of oxygen when heated to redheat. It is attacked by fluorine at room temperature and by chlorine and bromine at elevated temperature. It is attacked slowly by concentrated hydrofluoric acid and HNO_3 -HF mixture, but resistant to other mineral acids. It is also attacked by fused alkalis, but not by fused KHSO_4 .

ZIRCONIUM SILICIDE (ZrSi_2)

The disilicide ZrSi_2 is described as a gray powder and crystallizes in the form of rhombic pillars (Ref. 6). In compact form, the disilicide is stable in air at elevated temperatures. Except for hydrofluoric acid, mineral acids do not attack the disilicide. It is stable in alkali solutions, but is readily soluble in fused alkalis.

VANADIUM SILICIDE (VSi_2)

Vanadium disilicide, VSi_2 , crystallizes in the form of prisms with metallic luster. The disilicide is oxidation-resistant in air even at red heat. Except for hydrofluoric acid, mineral acids and aqueous solutions of alkalis do not attack VSi_2 . It is, however, readily decomposed by fused alkalis. The micro-hardness, using a 100-g load, is 1090 kg/mm².

TITANIUM SILICIDES (TiSi_2)

Titanium silicide, of the chemical formula TiSi_2 , crystallizes in iron-gray, flat tetragonal pyramids. Partly fused specimens obtained by synthesis at 3060 F are described by Brewer and co-workers (Ref. 6) as having a silver-blue metallic appearance.

Titanium disilicide oxidizes slowly at red heat in air. Mineral acids do not attack it, with the exception of hydrofluoric acid. Melting alkalis react violently with TiSi_2 .

MOLYBDENUM SILICIDES (MoSi_2 , Mo_3Si , Mo_3Si_2)

Three stable molybdenum silicides are generally recognized: Mo_3Si with 8.9-percent silicon; Mo_3Si_2 with 16.3-percent silicon; and MoSi_2 with 36.9-percent silicon.

The disilicide, MoSi_2 , is gray with a metallic luster, and is the most interesting of the various silicides. It has outstanding resistance to oxidation at elevated temperatures, and is not markedly attacked at red heat by a stream of oxygen. It reacts with fluorine at room temperature

and with chlorine at elevated temperature, but is not markedly attacked by a $\text{Br}_2\text{-H}_2$ stream at red heat. Molybdenum disilicide is resistant to mineral acids, including aqua regia and hydrofluoric acid, but is rapidly dissolved in a $\text{HNO}_3\text{-HF}$ mixture. It is decomposed by fused alkali, but resists fused KHSO_4 (Ref. 6).

The formula MoSi_2 apparently begins to decompose before melting, not only in the presence of carbon, but also when heated in stabilized zirconia crucibles (Ref. 16).

CHROMIUM SILICIDE (Cr_3Si , CrSi_2)

Chromium silicide, of the formula Cr_3Si , crystallizes in the form of prismatic crystals, and CrSi_2 crystallizes in gray needles of metallic luster. All chromium silicides are very stable toward mineral acids; only hydrofluoric acid dissolves them easily. Melting alkalis, however, dissolve them rapidly.

INTERMETALLICS

Intermetallic compounds have been known for a long time, and the properties of some of the modern super alloys are attributed to the presence of these compounds in a finely divided state. They are found in a large number of phase diagrams but their content in alloys has usually been carefully limited by the fabricators because they result in some detrimental properties, especially brittleness.

Some of the intermetallic compounds have very pronounced congruent melting points which are higher than the parent metals. This is very rare and seldom happens; however, since the number of high-melting metallic materials is rather limited, it appears desirable to perform a closer investigation of possible combinations to reveal their usefulness as high temperature materials.

Two intermetallic refractory alloy systems of current interest in the solid propulsion industry are the Mo-W and the Ta-W systems. Physical and mechanical properties for these two systems may be found in Tables 15 and 16.

MOLYBDENUM-TUNGSTEN ALLOY

Climelt molybdenum-30-percent tungsten alloy contains nominally 30-percent tungsten by volume. The alloy is designed mainly for ultra-high-temperature service under conditions where advantage can be taken of the fact that it has a higher melting point than unalloyed molybdenum.

The mechanical properties tabulated in Table 15 were obtained from an alloy of 28.91-percent tungsten and 0.025-percent carbon. Creep-rupture stress for the same alloy at 1800 F was 60,000 psi for a rupture time of 1 hour and 50,000 psi for a rupture time of 28.1 hours.

Climelt molybdenum-30-percent tungsten has better corrosion resistance than unalloyed moly in some mediums. It has been successfully extruded and forged. A satisfactory forging temperature is 2300 F. Results to date indicate that this alloy requires more reheating and higher working pressures than unalloyed molybdenum. The alloy is machinable by conventional and spark-discharge methods. Climelt molybdenum-30-percent tungsten is commercially available.

TANTALUM-TUNGSTEN ALLOY

Tantalum 90-percent, tungsten 10-percent alloy offers potential as a refractory material. The material shows good resistance to mechanical and thermal shock and has a high ductility at low temperatures; however, its use is limited by lack of resistance to chemical attack at metal part temperatures above 5000 F.

The recrystallization of the alloy does not appear to have any effect on the properties of the annealed material. Evidence to date indicates that annealed and or recrystallized sheet may be deformed extensively at room temperature without difficulty.

The 90-percent tantalum 10-percent tungsten alloy with an area reduction of 75 percent of the annealed specimen exhibits 125,000-psi ultimate strength.

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The forging temperature is between 2000 to 2200 F. The annealing temperature is 2200 F. The rolling temperature from forged plate is 800 to 1000 F. Cold-working of 90-percent reduction can be performed between annealing. The alloy may be inert atmosphere-welded in a dry box, and can withstand a conventional 90-deg bend test without failure. The alloy is available in ingot or sheet form. Tantalum 85-percent/tungsten 15-percent is stronger than 90-percent tantalum/10-percent tungsten at high temperature, but increasing the tungsten content introduces difficulty in fabrication. The material can be produced by either electron-beam or vacuum-arc melting, which produces material of equivalent purity and properties

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GRAPHITE

Graphite is a soft, slippery material having a grayish-black color. The powdered form is obtained by mining in which state it poses some purity and unfirmity problems and is referred to as natural graphite. Subjecting various carbon bodies to a temperature of approximately 4900 F results in very pure material referred to as artificial graphite.

The element carbon is generally found in an impure state and in abundance in many parts of the world. It exists in nature in the form of bituminous and anthracite coal, diamonds and natural graphite.

At ordinary temperatures, carbon is one of the most inactive of the elements. At high temperature, however, it becomes one of the most active. As transitional materials, they offer many of the properties found in both metals and non-metals, including inertness to chemical action, self-lubricating properties, high-heat conductivity, non-fusing properties, arc characteristics, and many others.

Some of the more common industrial sources of carbon are petroleum coke (obtained as the residue in the manufacture of gasoline), lampblack (obtained by the incomplete combustion of oil), and carbon blacks (obtained by thermal decomposition of gases). By combining these various raw materials with carbonaceous binders such as tars, pitches and resins, carbon forms and shapes may be produced by extruding or molding. The selection of the particular coke might depend on a number of considerations; however, the major reasons for their use are low cost, relatively low impurity content, and ease of graphitizing. These factors outweigh the disadvantages associated with a non-reproducible by-product raw material so far as the vast majority of the industrial market is

concerned. It would be beneficial to exercise more control over the raw materials, but this control is not currently compatible with the existing petroleum industry processes. The extruded or molded forms are slowly heated and then baked in the temperature range of 1400 to 1700 F. In the process of attaining this final temperature, the binder pyrolyzes evolving large quantities of gas. Heating must be slow to allow the gas to escape without distorting or breaking the piece. Polymerization and cross-linking in the binder and between the binder and filler occurs during the 1400 to 1700 F temperature baking. The final heat treatment of 4500 to 5400 F is then performed, which removes the remaining volatiles and converts the carbon to graphite.

By controlling the various intermediate processing techniques during the baking and graphitizing cycles, graphite with the desired properties may be obtained. For example, high strength and more impermeable graphite may be obtained by impregnating the form after the baking process with more binder and then rebaking before proceeding into the graphitizing cycle. Properties of graphite may also be controlled during graphitization, resulting in thermal expansion properties compatible to the properties of various coating materials.

Commercial graphite has a fairly low density (1.6 to 1.7 gm cm^3); it is hard compared to natural graphite flakes: it is a strong material, and at temperatures above 2700 F, it is one of the strongest known. Commercial graphite is porous to gases having variable properties from piece to piece and also within a piece, because of the nature of the process used to make it. The porosity of the graphite can be divided into two types:

- (1) pores that are left due to the loss of volatile constituents, and
- (2) those that are caused by differential contraction of the crystallites.

The voids caused by the volatile constituents of the binder constitute

the major portion of the porosity in commercial graphite. These voids may be reduced by successive impregnations following the baking cycle, but theoretically cannot be processed out. The second type of voids are caused by differences in contraction along the A and C directions during the cooling cycle. These are unavoidable voids but may be controlled by the orientation of the crystallites. These voids are practically eliminated in graphite bodies of highly preferred orientation.

Pyrolytic graphite, a new custom-made form of graphite, is being developed for the rocket industry as a result of its exceptional properties. This form of graphite exhibits a high degree of preferred orientation and a wide range of anisotropy in thermal and electrical properties. It is a polycrystalline graphite formed by high-temperature pyrolysis of a carbon-bearing material. It is currently manufactured by a process of depositing carbon in graphite form from a carbonaceous gas on a mandrel. The carbon is derived from thermal decomposition of the gas. Through control of processing variables, such as pressure, gas flow, and furnace temperature, graphite crystallines of varying order and orientation can be prepared. The resulting material properties vary from nearly isotropic to highly anisotropic. For example, in the highly anisotropic case, thermal conductivity in the layer planes A direction is in the order of 200 times greater than the thermal conductivity in the normal or C direction.

Pyrolytic graphite is readily finished by standard machine and hand tools. A preference has been shown for diamond or carbide-tipped grinding tools since cutting tools tend to delaminate the material. Pyrolytic graphite will reproduce the mandrel surface finish on the face adjacent to the mandrel. The final deposited surface is about 200 to 300 rms. Surface finishes of 4 to 6 rms can be obtained by polishing.

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Pryolytic graphite is stronger than commercial graphite, and near room temperature the thermal conductivity along the basal planes is higher than that of any known material, while at slightly higher temperature, the thermal conductivity perpendicular to the layer planes is lower than that for any other solid thermal insulator.

For room temperature, mechanical and physical properties of various graphites, see Tables 17 and 18 . Some elevated temperature properties are disclosed in Fig. 19 through 24.

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CONVERSIONS OF UNITS FOR COMMONLY USED PROPERTIES

Property	Factor x Unit	Equivalent Unit
Density	27.68 lb/in. ³	gm/cm ³
Thermal conductivity	241.9 gm cal/cm ² sec C per cm	Btu/hr ft ² F per ft
Temperature	1.8 C + 32	F
Unit per degree	1.8 unit/C	unit/F
Load/unit area	0.000703 kg/mm ²	lb/in. ²

Various tables showing comparative hardness scales may be found in the Machinery Handbook. A brief description of the tests for the most commonly used scales is given below.

BRINELL HARDNESS SCALE

The Brinell test for determining the hardness of metallic materials consist of applying a known load to the surface of the material to be tested through a hardened steel ball of known diameter.

$$\text{Brinell Number} = \frac{\text{load on indenting tool in kg}}{\text{surface area of indentation in mm}^2}$$

VICKERS HARDNESS SCALE

The Vickers hardness test is similar in principle to the Brinell hardness test. The standard Vickers penetrator is a square-based diamond pyramid having an included point angle of 136 deg. The numerical value of the hardness number equals the applied load in kg divided by the area of the pyramidal impression.

KNOOPS HARDNESS NUMBER (KNH)

The Knoop hardness tester is applicable to extremely thin metal, plated surfaces, exceptionally hard and brittle materials, very shallow carburized or nitrided surfaces, or whenever the load must be kept below 3600 gm. The Knoop hardness number equals the load in kg divided by the projected area of indentation in mm^2 .

MOHS'S HARDNESS SCALE

Hardness, in general, is determined by what is known as Mohs's scale, a standard for hardness which is mainly applied to nonmetallic elements and minerals. In this hardness scale, there are ten degrees or steps, each designated by a mineral, the difference in hardness of the different steps being determined by the fact that any member in the series will scratch any of the preceding members. This scale is as follows: (1) talc, (2) gypsum, (3) calcite, (4) fluor spar, (5) apatite, (6) orthoclase, (7) quartz, (8) topaz, (9) sapphire or corundum, and (10) diamond. These minerals, arbitrarily selected as standards, are successively harder, from talc, the softest of all minerals, to diamond, the hardest. This scale, which is now universally used for nonmetallic minerals, is, however, not applied to metals.

ROCKWELL HARDNESS TEST

The Rockwell hardness tester is essentially a machine that measures hardness by determining the depth of penetration of a penetrator into the specimen under certain fixed conditions of test. The various Rockwell scales and their applications are shown below.

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Rockwell Scale

Testing Application

A	For tungsten carbide and other extremely hard materials. Also for thin hard sheets.
B	For materials of medium hardness such as low and medium carbide steels in the annealed condition.
C	For materials harder than Rockwell B-100.
D	Where somewhat lighter load is desired than on C scale, as on case-hardened pieces.
E	For very soft materials such as bearing materials.
F	Same as E scale but using a 1/16-in.-dia ball.
G	For metals harder than tested on B scale.
H & K	For softer metals.
15-N, 30-N; 45-N	Where shallow impression or small area is desired. For hardened steel and hard alloys.
15-T; 30-T; 45-T	Where shallow impression or small area is desired for materials softer than hardened steel.

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HIGH TEMPERATURE PROPERTIES

Very limited information is available for high-temperature properties of refractory materials. This section shall consist of graphs representing the effects of temperature on the properties of some of the refractory materials. The curves (Fig. 1 through 25) are intended to represent the properties of the subject materials more as a trend rather than concise design values, which vary depending on the purity of the material and the method of fabrication.

MATERIAL PRODUCERS AND FABRICATORS

The nozzle throat materials for solid rocket motors have to withstand some of the most severe conditions that materials can be subjected to.

They must withstand high temperatures, thermal shock, chemical attack by combustion products, and erosion. Further, in order to satisfy performance requirements, the nozzle throat material must be machined to close tolerance with smooth, non-wavy surfaces. The material must have consistency in properties and, in most cases, must be free of impurities. Impurities found in some metals of present commercial use might easily prevent a usable material from performing satisfactorily.

The materials which will perform satisfactorily under the existing conditions are recently new to commercial material suppliers and fabricators and, due to the quality of the material required, special care must be taken in their production. In cases of complicated shapes and normally hard-to-fabricate materials, special fabrication methods are being developed.

Material suppliers and fabricators, and methods of fabrication for some of the materials currently used in rocket nozzles are briefly discussed below. Unless the material supplier is specifically called out, the fabricators have their own source of raw materials. Primary producers of refractory metals may be found in Table 19.

TUNGSTEN

Forms of tungsten are being produced by the following:

1. Forged tungsten, material supplied by Allison Division of General Motors Corp., then sintered, billet-forged, and machined by Ladish Company
2. Solid tungsten, supplied and arc-cast by Firth Sterling Company
3. Vapor-deposited tungsten, in the form of a ring, produced by American Metal Products Company
4. Tungsten wire wrap on graphite, supplied by Thermionic Corp. The tungsten wire (5 mil), wrapped on a small graphite ring
5. Sintered tungsten, sintered and machined by Sintercast Corp.
6. Plating of metals by vapor deposition of high-purity tungsten have been developed by the National Bureau of Standards and by the Bureau of Mines. Adhesive bonds are rated good to nickel and molybdenum, weaker to copper; poor to iron. Tungsten-graphite bond is as strong as graphite itself.
7. Tungsten ingots, 10-in. dia x 4 ft long in rectangular, cylindrical, or hollow cylinder shapes are supplied by Sylvania Electric Products, Inc., Chemical and Metallurgical Division.
8. Plasma-arc-sprayed tungsten coatings on graphite or free-standing material are being fabricated in many complex shapes by Linde Co., Division of Union Carbide Co. Tungsten coatings of 0.040 in. on a tantalum undercoat of 0.003 in. have been produced on graphite base. The porosity of this material is increased by heat treating. The detrimental effect of tungsten carbide appearing during heat

treating does not occur due to the tantalum undercoat. A further discussion of Lindes arc-spray process is presented in the section titled Fabrication Techniques.

9. Micrograin tungsten ingot, supplied by Stauffer-Temescal, and forged into an 8-in. nozzle by Ladish Company.
10. High-density tungsten vapor deposited on graphite 1/8-in. thick is produced by Alloyd Corp., Cambridge, Mass. This process involves hydrogen reduction of tungsten hexachloride. This material is also being produced by High Temperature Materials, Inc.

MOLYBDENUM

Forms of molybdenum are being produced by the following:

1. Forged molybdenum, material supplied by Steel Improvement and Forge Company. The metal is arc-cast, billet-forged, and machined by Custom Machine Company.
2. Solid molybdenum supplied, arc-cast, and machined by Climax Molybdenum Company
3. Arc-cast molybdenum, supplied by Climax Molybdenum Company and machined into a nozzle insert ring by Thiokol Chemical Corp.
4. Micrograin molybdenum ingots are produced by Stauffer-Temescal
5. Refractomet, Division of Universal-Cyclops has produced very large molybdenum sheet.

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GRAPHITE

Forms of graphite are being produced by the following:

1. Various grades of graphite have been supplied by National Carbon Co., American Metal Products Company, Crescent Carbon Company, Great Lakes Carbon Company, Speer Carbon Company, Pure Carbon Company, Stackpole Carbon Company, Morganite Carbon Company, and U. S. Graphite Company.
2. High Temperature Materials, Inc. in Boston is producing high-quality pyrolytic graphite in complex shapes and plate forms. The material is deposited in thicknesses up to 3 8 in. They are capable of producing free-standing pieces 6 ft long and 30 in. in diameter.
3. National Carbon Company has extruded graphite and designated it commercially CS graphite. They also produce a grade ZTA with a density of 1.95 g/cm^3 and ATJ grade of density 1.73 g/cm^3 . The purpose of the program is to reduce the variation in materials and the time required for processing and fabricating materials into "hot" components.
4. American Metal Products Company has molded graphite by a proprietary process and designated it, commercially, HD graphite. Various grades of graphite are produced being impregnated at high temperatures.
5. Raytheon Company, Waltham, Mass., has put high-purity graphite (pyrographite) on the commercial market. It withstands temperatures up to 6700 F--remaining strong, chemically inert,

and impermeable to gases. Oriented graphite may be combined with metals for special alloys. The Navy's Special Products Office, Bureau of Naval Weapons, is examining samples.

6. Edison's pyrolytic graphite maintains strength up to 6500 F with thermal conductivity 1000 times greater in one direction than the other.
7. Great Lakes Carbon Company's methods of producing graphite include: stock extruded, retreated with pitch, and regraphitizing; and molded shapes.

INTERMETALLIC COMPOUNDS

Various intermetallic compounds are being produced by the following:

1. Forged 50W-50Mo alloy, forged and machined by the Landish Company.
2. Tungsten-molybdenum alloys of 50W-50Mo, 70W-30Mo, 60W-40Mo, and 85W-15Mo, supplied by Oregon Metallurgical Corporation. The tungsten-molybdenum alloys of 50W-50Mo and 70W-30Mo are arc-cast and machined to thin-ring configuration, and 60W-40Mo and 85W-15Mo are arc-cast and machined to rings with tapered ends by the Thiokol Chemical Corporation.
3. Forged 30W-70Mo and 50W-50Mo alloy rings, sintered, forged, and machined by the Steel Improvement and Forge Company.
4. Arc-cast tungsten-molybdenum alloys of 50W-50Mo, 60W-40Mo, 70W-30Mo, and 85W-15Mo were double arc-melted and machined by the Oregon Metallurgical Corporation.

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5. Electron beam melted tantalum-tungsten alloy of 90Ta-10W, supplied by Stauffer Metals Company and machined by Rocketdyne.
6. Electron beam melted molybdenum-tungsten alloy of 70Mo-30W supplied by Stauffer Metals Company and machined by Rocketdyne.
7. Tantalum-tungsten alloy of 90Ta-10W, supplied by Tremescal. Electron beam melted alloy, forged and machined by California General
8. High-temperature alloys are being commercially produced by Haynes Stallite Company, Carboloy Division of General Electric, International Nickel Company, Universal Cyclops Corp., and Coast Metal, Inc.

CARBIDES AND CARBIDE COMPOUNDS

Various carbides and compounds are being produced by the following:

1. Columbium carbide and tantalum carbide, Firth Sterling, Inc.
2. Tantalum carbide, Electro-Thermal Industries
3. Columbium carbide-tungsten, NbC powder pressed with 10-percent tungsten binder and sintered by Sintercast Corporation
4. Zirconium carbide-tungsten, ZrC powder pressed with 10-percent tungsten binder and sintered by Sintercast Corporation
5. Hafnium carbide, tungsten, HfC powder pressed with 10-percent tungsten binder and sintered by Sintercast Corporation
6. Tantalum carbide-tungsten, TaC powder pressed with 10-percent tungsten as binder and sintered by Sintercast Corporation
7. Titanium carbide-tungsten, TiC powder pressed with 10-percent tungsten as binder and sintered by Sintercast Corporation

8. Tantalum carbide-tantalum, material supplied by National Research Corporation, and liners formed, carburized, and backed with carbon by the American Metal Products Company
9. Rhenium-carbide, impregnation of ammonium per-rhenate in graphite and drying reduction by University of Utah
10. Tungsten carbide, sintered and ground tungsten carbide with 6-percent cobalt binder by Shwayder Chemical and Metallurgical Corporation
11. Silicon carbide bonded on graphite, the composite SiC and graphite sintered and machined by Carborundum Company
12. TaC-Ta₂C-Ta, tantalum carbide coating impregnated on tantalum liner by American Metal Products Company
13. TaC-Ta₂C-C, tantalum carbide coating impregnated on HD graphite by American Metal Products Company
14. TaC-HfC-C, HD graphite impregnated with tantalum carbide and hafnium carbide at high temperatures by American Metal Products Company
15. TaC-BeC-C, impregnated at high temperatures by American Metal Products Company
16. SiC-TiC-C, siliconized titanium carbide, impregnated on HD graphite by American Metal Products Company
17. TiB₂-TiC-C, titanium diboride, impregnated on HD graphite by American Metal Products Company
18. Titanium carbide, reinforced with tungsten fiber by Amour Research Foundation
19. Some other companies having experience with carbides include Norton Company; Welded Carbides, Inc.; Carboloy Division, General Electric; American Electro Metal Company; and Linde, Division of Union Carbide.

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20. Hafnium carbide, research is being performed by NASA's Lewis Research Center.
21. Union Carbide Metals Company is studying borides and carbides of Ti, V, Nb, Ta, Cr, W, Hf, Mo, and Zr.
22. NbC, TaC, and WC have been pyrolytically deposited on graphite in thicknesses up to 50 mils by High Temperature Materials, Inc.

OXIDE COMPOUNDS

Various oxide compounds are being produced by the following:

1. Tungsten-beryllium oxide, pressed and sintered by powder metallurgy techniques (cermet 2410-185) by Bendix Aviation Corporation
2. W-BeO-Cu cermet, powdered, pressed, and sintered by Bendix Aviation Corporation
3. Mo-BeO-Cu, molybdenum and beryllium oxide powder, pressed and sintered and impregnated with copper by the Bendix Experimental Laboratories
4. Mo-BeO-Cu-MoO₃, powder pressed and sintered by Bendix Experimental Laboratories
5. ZrO₂-C material supplied by the National Lead Company and the National Carbon Company; zirconia sintered onto graphite by the University of Utah
6. W-Al₂O₃ (cermet 3870-1), produced by Bendix Aviation Corp. It has a compressive strength of 140,000 psi, a hardness of Rc30, and a density of 14.6 gm/cm³
7. Cermets, several cermet impingement samples have been fabricated by Technical Research Group, Inc., by hot-pressing techniques.

REFRACTORY MATERIAL COATINGS

Various refractory material coatings are being applied by the following:

1. Tungsten-rhenium ring, tungsten ring was forged, machined, and sprayed with rhenium by Allison Division of General Motors Corp.
2. Tungsten-rhenium-graphite, rhenium is sprayed on graphite in a thin coating to prevent the tungsten, which was sprayed over the rhenium, from reacting with the graphite. Coating applied by Linde Division of Union Carbide.
3. Tungsten coatings on graphite, vapor deposition with no undercoat by the U.S. Bureau of Mines and High Temperature Materials, Inc.
4. Plasma arc-sprayed coatings, available from Linde Division of Union Carbide, include refractory oxides and carbides, zirconia, alumina, molybdenum, tungsten.

MISCELLANEOUS

1. Metcut Research Association, Inc., Cincinnati 9, Ohio, currently has a government contract for developing machining techniques of advanced materials, which should last approximately 2 years.
2. Ductile refractory metals are being made available in single crystalline form by Materials Research Corporation, Yonkers, New York, and by Linde Division of Union Carbide.
3. Porous metals (stainless steel and high temperature metals), shapes as well as flat stock, are available with "unprecedented" control over pore size, distribution, ductility, permeability, and other properties by Mott Metallurgical Corporation, Hartford, Connecticut

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4. Ceramics, nickel substituted for part of the iron in the binder of tungsten carbides to improve the strength and hardness of carbide compacts, is being performed by the Army's Watertown Arsenal, Watertown, Mass.
5. The National Carbon Company, Division of Union Carbide, has a \$12 million research program in conjunction with graphite and graphite base materials.
6. High Temperature Materials, Inc. has pyrolytically deposited small percentages of B, Al, W, and Nb with pyrolytic graphite. A 1-percent boron addition to the pyrolytic graphite has been shown to increase the room temperature strength of pyrolytic graphite appreciably. This company has also successfully pyrolytically deposited boron nitride.

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FABRICATION TECHNIQUES

The many design problems encountered while designing solid rocket motors with the extreme requirements involved, have brought about many new techniques of fabrication--some methods becoming necessary for the lack of a better means of fabrication and others due to specific design requirements.

As a result of the problems encountered in fabrication, new and older but more sophisticated fabrication techniques are being developed. As an example, the ability to melt refractory materials such as tungsten and molybdenum then deposit them as very dense, high-purity coatings on a variety of substrates, opens a new manufacturing technique for these normally hard-to-fabricate materials.

Three methods of successfully fabricating refractory materials of complex shapes are explosive compaction, plasma arc-spraying, and vapor deposition. In addition to free-standing bodies, the latter two methods are used for producing coatings of some refractory materials on various substrates. In addition to explosive forming, another high-energy compaction process is under development by the Precision Forge Company of Santa Monica, California. This process involves high-energy forging utilizing a Dynapak machine and looks very promising for hard-to-fabricate shapes.

DYNAPAK HIGH-ENERGY FORGING

Dynapak is a high-energy forging machine which incorporates a ram traveling 1000 in./sec, permitting instantaneous fabrication of complex shapes.

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High-energy rate machines like Dynapak take advantage of the plastic behavior of hard, tough, or brittle metal when they are hit with abnormally high-pressure flows. Taking advantage of this plastic behavior under the conditions resulting from the Dynapak machine offers great promise in forging hard-to-fabricate materials of complex shapes encountered in advanced solid propellant motors.

Compared with conventional methods, Dynapak makes possible closer tolerances, thinner sections, finer surface finishes, smaller radii, and occasionally, better metallurgical properties.

EXPLOSIVE FORMING

Explosive compaction may have a great future with refractory materials. Certain refractories, such as tungsten, due to the nature of their crystalline structure, are particularly responsive to shock or impact forming, whereas under normal deformation procedures, cracking and brittle fracture occur.

Complex nozzle shapes may be formed by explosive compaction of powdered tungsten, molybdenum, tantalum, and columbium, giving excellent forms. The most easily compacted refractory metal is expected to be columbium, the density of which is less than half that of tungsten, but the melting point of which is too low for use as an unprotected nozzle component.

Coating materials by explosive forming techniques and the fabrication of cermets by explosive compaction of metal and ceramic powder combinations appear possible, but to date require further investigation.

Materials compacted at Rocketdyne include graphite, boron, tungsten, Al_2O_3 , moly sulfide, lithium hydride, and polymers. These materials have been compacted by the explosive process to densities in the 97 to 100-percent theoretical range. The work done at Rocketdyne has been on a limited fund basis and, accordingly, was limited in scope. In each case listed, it was shown that a solid, essentially void-free, piece of the material could be formed. It is believed that this process has great capability and deserves further development.

PLASMA-ARC SPRAYING

Plasma arc-spraying has opened the door for many refractory materials which could not be easily fabricated due to their hardness and high-melting points. The plasma arc-spraying process makes use of a torch which produces and controls a high-velocity gas stream that can be maintained at temperatures above 20,000 F for periods well over an hour. The hot gas stream can melt and accelerate, to high velocities, particles of any known solid inorganic material which will melt without decomposition. When these molten materials strike the work piece, they impact to form a dense, high-purity coating. Free-standing hardware may be produced by utilizing a removable mandrel technique, in which the work piece is removed; the material may then be densified by a sintering-type heat treatment and machined to the final dimension.

Since at the present time tungsten offers the most desirable properties of the refractory metals, the plasma arc-spraying process will be further described using the fabrication of tungsten products for a means of discussion.

Conventional air-sprayed materials are contaminated with oxygen and nitrogen. Ninety percent of the contamination in tungsten, when spraying in air, may be removed by sintering; however, the impurity level is still slightly higher than commercial tungsten. Free-standing tungsten hardware made by the removable mandrel technique may be densified by a sintering, resulting in a fine-grained equiaxed microstructure. The structure is very similar to cold-pressed and sintered material. Heat treatment removes all traces of the lamellar structure due, mainly, to varying levels of oxygen impurity incurred during the spray operation. When spraying in an inert atmosphere, the contamination is decreased and the density is increased. The density of inert-atmosphere-sprayed tungsten is equivalent to conventional-sprayed and heat-treated tungsten. Some comparative room temperature properties of tungsten and arc-sprayed tungsten are shown in Table 20 and Fig. 26. Tensile properties of sintered, arc-sprayed tungsten are compared with those of commercial-wrought rod and recrystallized commercial rod in Fig. 28. The arc-sprayed material is quite similar to the recrystallized wrought rod.

The ductile-brittle transition zone for sintered arc-sprayed tungsten was found to be from 600 to 650 F. For comparative purposes, the bend ductility of commercial-wrought recrystallized tungsten is plotted in Fig. 27. The very close proximity of the ductile-brittle transition zone with the arc-sprayed material is apparent.

The density of arc-sprayed tungsten varies over the range of 85 to 90 percent of the theoretical density with an ultimate tensile strength of approximately 20,000 to 30,000 psi.

Both Linde and Allison have developed removable mandrel techniques for fabricating free-standing bodies. Arc-spray and sintering techniques

of fabricating free-standing shapes offer no size limitation other than a minimum throat size, low tooling cost, flexibility of design contours, and consistency from part to part.

Allison has produced tungsten with wall thicknesses greater than 1 in. and weighing over 100 lb. Their full-scale mandrels are permanent in the sense that they can be used to make a number of pieces of hardware. The mandrel is so constructed that cracking of the tungsten during buildup is prevented.

Linde's process is somewhat different and is considered a lost mandrel process. Tungsten is sprayed on a brass mandrel which is then etched out. This technique will allow very complex parts to be readily fabricated. Linde reports a higher density than Allison, having a hoop tensile strength of 60,000 psi and a tensile strength of 30,000 psi. Heat treatment of plasma arc-sprayed tungsten, prepared by the lost mandrel technique, will decrease in over-all size. Linde reports that the theoretical density may be increased to 94 percent and with additional treatment of a special type to 97 percent. Also, the physical properties may be enhanced by 50 percent. The modulus of elasticity will be increased at a greater rate than the modulus of rupture which is very advantageous.

Both sintered arc-sprayed tungsten and cold-pressed and sintered tungsten may be forged without internal or edge cracking. The development of tungsten forgings, using sprayed preforms, appears promising and offers the potential advantages of greater strength and toughness. To date, forging has increased the strength of arc-sprayed tungsten, but only at the expense of ductility, the ductile-brittle transition temperature being elevated. The ductility of forged material may be improved by increasing the purity of the preforms. In the case of preforms made

from arc-sprayed tungsten, the purity as stated before may be markedly increased by controlled atmosphere spraying and additional heat treatment if necessary.

Direct arc-spray coatings on work pieces, which become an integral portion of the final product, have many advantages over free-standing materials. They are cheaper and can be applied in relatively thin thicknesses and still maintain structural stability, thereby allowing light coatings to be applied. The use of sintering as a method of improving coating properties is prevented in cases where sintering shrinkage varies with the coating and the base material, and when the reaction of the coating with the base material is detrimental to the material. For instance, when heat treating a tungsten coating on a graphite base, tungsten carbide is formed resulting in a poor brittle bond. Controlled atmosphere spraying with the present knowledge appears to be the best method of improving coating properties. However, with more knowledge, concerning the reaction effects of various materials, it may be possible to spray very thin layers of intermediate materials between the coating and the base material, which may be heat-treated without detrimental effects. They may also provide better bonds, ductility, and over-all properties than those which presently exist.

VAPOR DEPOSITION

Vapor deposition produces coatings with high penetration on parts of any size or shape. It is a comparatively fast method and can be performed in a continuous operation. Vapor deposition is accomplished by forcing a plating gas containing a volatilized compound of metal to be deposited past the heated base metal in a partially evacuated system. The actual process is relatively straightforward, but considerable engineering know-how is needed for efficient operation.

High-purity tungsten coatings, vapor-deposited from tungsten hexafluoride, produce smooth, fine-grained and bright deposits. The coating exhibits an adherent mechanical bond between the graphite and tungsten.

Vapor-deposited tungsten has exhibited excellent stability in solid rocket motors, and to date, is standing up better than plasma arc-sprayed coatings applied on graphite bases, probably due to the penetration of the tungsten establishing a more rigid bond. The tungsten graphite bond is as strong as the graphite itself.

The Bureau of Mines has developed a low-temperature vapor-deposition method of producing 99.99-percent-pure tungsten and depositing it in readily formed shapes.

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TABLE 1
ROOM TEMPERATURE TENSILE PROPERTIES OF VANADIUM OF DIFFERENT
PURITIES PRODUCED BY DIFFERENT METHODS

Specimen Number	Chemical Analysis			Yield Point	Ultimate Tensile Strength, psi	Elongation, percent	Reduction in Area, percent
	O ₂	N ₂	C				
1	0.012	0.008		IODIDE VANADIUM 15,300 (0.1) 16,700 (0.2)	31,600	17	75
CALCIUM REDUCED VANADIUM ARC MELTED AND FORGED							
2	0.08	0.04	0.05	42,000	55,000	32	72
3	0.07	0.04	0.07	55,000	63,600	28	72
4	0.08	0.04	0.07	63,000	69,600	32	66
5	0.06	0.04	0.09	56,000	63,600	36	68
6	0.10	0.05	0.06	65,000	72,200	32	67
7	0.08	0.05	0.06	52,000	60,300	34	69
8	0.10	0.05	0.06	64,000	66,400	38	82
9	0.09	0.07	0.10	64,000	74,600	26	68
10	0.15	0.08	0.14		71,900	28	
11	0.10	0.15	0.15		74,500	24	
12	0.18	0.10	0.12		40,000	0	0
13	0.09	0.08	0.25		83,900	10	25

TABLE I
Continued

Specimen Number	Chemical Analysis			Yield Point	Ultimate Tensile Strength, psi	Elongation, percent	Reduction in Area, percent
	O ₂	N ₂	C				
				CALCIUM REDUCED VANADIUM EXTRUDED			
14	0.045	0.007	0.13	57,800 {upper}	61,500	35	64
15	0.031	0.026	0.146	51,700 {upper}	63,600	31	56
				48,900 {lower}			
16	0.032	0.001	0.075	47,200 {upper}	60,800	31.5	70.5
				44,300 {lower}			
				POWDER COMPACT			
17	0.057	0.071	0.09	53,700	60,600	18.9	

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TABLE 2

PHYSICAL PROPERTIES OF REFRACTORY METALS AT ROOM TEMPERATURES

Material	Melting Temperature,		Boiling Temperature,		Density		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion, F x 10 ⁶	Coefficient of Thermal Conductivity,	
	C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Tungsten	3410	6170	5900*	10630*	19.3	0.697	0.034	2.2	93.1*	0.385*
Rhenium	3170	5740	5650	10200	21.0	0.759		2.8	41.0	0.170
Tantalum	2996	5413	5000	9000	16.6	0.600	0.076	3.6	71.5	0.130
Osmium	2700	4890	5000	9050	22.5	0.82	0.031	3.6		
Molybdenum	2620	4748	3700	6710	10.2	0.368	0.065	5.3	84.5	0.350
Columbium	2500	4530	3500*	5960*	8.56	0.309	0.065	3.9	30.3	0.125
Iridium	2450	4440	3500	9570	22.4	0.809	0.031	3.8	84.6	0.350
Ruthenium	2430	4410	4000	8850	12.2	0.441	0.057	5.1		
Hafnium	1975	3585	3400	9735	13.0*	0.470*	0.035	3.4	12.8	0.035
Rhodium	1970	3550	4500	8130	12.4	0.448	0.059	4.6	87.0	0.360
Vanadium	1900	3450	3550	6050	6.11*	0.220*	0.120	4.8	17.9-21.3	0.074-0.088
Chromium	1870	3417	2649	4480	7.2	0.260		3.5	38.7	0.16
Zirconium	1846*	3355*	4570	7920	6.4	0.231	0.069	3.1*	9.6	0.040
Thorium	1830	3330	3000	5430	11.7	0.423	0.030	6.2	21.4	0.088
Platinum	1780	3240	4300	7760	21.4	0.775	0.031	4.9	19.4	0.080
Titanium	1730	3150	3290	5940	4.5	0.163	0.125	5.2	10.4	0.043
Palladium	1560	2840	2200	3990	12.0	0.434	0.058	6.5	44.0	0.182
Beryllium	1175	2341	2480	4490	1.77	0.064	0.450*	7.0*	60.0*	0.248*
Uranium	1130	2070	3500	5680	19.1	0.690	0.028		14.5	0.060
Aluminum	660	1410	2057	3750	2.70	0.098	0.211	13.0	128.0	0.530

Note: Generally, refractory metals are considered to have a melting point at least greater than 1800 C; however, because of the importance of some lower melting point metals in compounds making up some refractory materials, they have been mentioned.

*Discrepancy in reported value.

TABLE 3
MECHANICAL PROPERTIES OF REFRACTORY METALS
AT ROOM TEMPERATURE

Material		Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness
Tungsten	W	50	70-500		C37-E47
Rhenium	Re	67.5	168-322	135-311	
Tantalum	Ta	27	40-180	39	E60-E95
Osmium	Os	80			Brinell 350
Molybdenum	Mo	47	65-175	50-100	C22-B95
Columbium	Cb	15	50-100	41-78	E80-E100
Iridium	Ir	74			Brinell 163-350
Ruthenium	Ru	60			Brinell 220
Hafnium	Hf	20	77-112	32-96	A58-A65
Rhodium	Rh	42	73-300		Brinell 155-390
Vanadium	V	20	55-110	40-109	B81-B93
Chromium	Cr	42	15-112		
Zirconium	Zr	14	24-90	11-84	B42-B98
Thorium	Th	10	34-49	26-35	
Platinum	Pt	21	17-45	2-27	Brinell 38-130
Titanium	Ti	16	50-110	40-95	
Palladium	Pd	17	30-47	5-30	Brinell 46-106
Beryllium	Be	44	35-90	30-45	
Uranium	U	30	90	25	
Aluminum	Al	10	8		Mohs 2

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TABLE 4
PHYSICAL PROPERTIES OF REFRACTORY CARBIDES AT ROOM TEMPERATURES

Carbides Melting Point Greater Than 1800 C		Melting Temperature,		Boiling Temperature,		Density		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion, F x 10 ⁻⁶	Coefficient of Thermal Conductivity,	
		C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Hafnium	HfC	3890	7030	4980- 5540	9000- 10000	12.2	0.442		4.6	9.8	0.053
Tantalum	TaC	3880	7020			14.39	0.520				
Zirconium	ZrC	3400	6150			15.1	0.545		3.85	11.8	0.049
Columbium	CrC	3530	6350	5100	9220	6.7	0.242		3.79	8.2	0.034
Titanium	TiC	3500	6300			7.82	0.282		4.22	9.9	0.041
Vanadium	VC	3150	5700	4300	7770	4.7	0.170	0.20			
		2830	5130	3900	7060	5.36	0.194				
Silicon	SiC	2830	5130	*	*	3.17	0.115	0.23	2.39-2.50	18.0	0.075
Aluminum	Al ₄ C ₃	2800	5070	**	**	2.95	0.107				
Tungsten	WC	2730	4950	6000	11630	17.2	0.621		3.28		
	W ₂ C	2630	4770	6000	11630	15.8	0.571		3.39		
Molybdenum	MoC	2695	4880			8.4	0.304				
	Mo ₂ C	2690	4870			8.9	0.322				
Thorium	ThC ₂	2655	4810	5000	9030	9.6	0.347				
	ThC	2625	4755			10.7	0.387				
Boron	B ₄ C	2450	4440	3810	6885	2.49	0.90	0.44	2.50	16.9-48.4	0.07-0.20
Uranium	UC ₂	2450	4440	4371	7900	11.3	0.408		6.95		
	UC	2350	4225			13.6	0.491		5.5	14.51	0.06
Beryllium	Be ₂ C	2145	3890			2.25	0.081		5.83	12.2	0.05
Chromium	Cr ₃ C ₂	1895	3440		4352	5.6	0.202		5.50		

* Decomposes 2200 C

** Decomposes greater than 2200 C

TABLE 5
MECHANICAL PROPERTIES OF REFRACTORY CARBIDES
AT ROOM TEMPERATURE

Carbides Melting Point Greater Than 1800 C		Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness
Hafnium	HfC				2900 Kg/mm^2
Tantalum	TaC	41.5	31		1800 Kg/mm^2 Mohs 9-10
	Ta ₂ C				1000 Kg/mm^2
Zirconium	ZrC	47	16		2600 Kg/mm^2 Mohs 8-10
Columbium	CbC	49			2400 Kg/mm^2
Titanium	TiC	45	48		3200 Kg/mm^2 Mohs 9-10
Vanadium	VC	39			2800 Kg/mm^2
Silicon	SiC	40	18		2480 Kg/mm^2
Aluminum	Al ₄ C ₃				
Tungsten	W ₂ C	60.5			3000 Kg/mm^2 Mohs 9
	WC	102	90		2400 Kg/mm^2 Mohs 9
Molybdenum	MoC				Mohs 7-9
	Mo ₂ C	32.7			1800 Kg/mm^2 Mohs 7
Thorium	ThC ₂				
	ThC				
Boron	B ₄ C	48	27		2750 Kg/mm^2
Uranium	UC ₂				
	UC				
Beryllium	Be ₂ C	46			2410 Kg/mm^2
Chromium	Cr ₃ C ₂		32		1300 Kg/mm^2

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TABLE 6
PHYSICAL PROPERTIES OF REFRACTORY NITRIDES AT ROOM TEMPERATURES

Nitrides		Melting Temperature,		Boiling Temperature,		Density $\mu\text{m cm}^{-3}$ lb./in. ³	Specific Heat, Btu/lb./F or Cal/gm/C	Coefficient of Thermal Expansion, F $\times 10^{-6}$	Coefficient of Thermal Conductivity,	
		C	F	C	F				Btu/hr/ft/F	Cal/cm/sec/C
Hafnium	HfN	3310	5990			14.0	0.506			
Tantalum	TaN	3100	5610			14.1	0.510			
Boron	BN	3000	5430	(1)	(1)	2.25	0.081	2.78-5.55	9.68-29.0	0.040-0.12
Zirconium	ZrN	2980	5400			7.32	0.264	3.33-3.88	9.68	0.040
Titanium	TiN	2930	5310			5.43	0.196		2.66	0.011
Uranium	UN	2650	4800			14.5	0.517			
Thorium	ThN	2630	4770			11.5	0.415			
Beryllium	Th ₃ N ₄	2360	4280			10.4	0.376			
Columbium	Be ₃ N ₂	2200	3990	(1)	(1)	8.4	0.303	2.24-3.38		
	CbN	2050	3720			8.08	0.292			
Vanadium	Cb ₂ N					6.04	0.218			
Silicon	VN	2050	3720	(2)	(2)	3.4	0.124	1.37	10.9	0.045
Chromium	Si ₃ N ₄	1900	3450			6.1	0.221			
	CrN	1500	2730							
Aluminum	Cr ₂ N			(2)	(2)	3.05	0.110		11.6-17.4	0.048-0.072
	AlN	2400	4350							

(1) Decomposes
(2) Sublimes

TABLE 7
MECHANICAL PROPERTIES OF REFRACTORY NITRIDES
AT ROOM TEMPERATURES

Nitrides		Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness
Hafnium	HfN	12.4			Mohs 8
Tantalum	TaN				Mohs 2 or 100 kg/mm^2
Boron	BN				Mohs 8 or 1510 kg/mm^2
Zirconium	ZrN				8000 kg/mm^2
Titanium	TiN				
Uranium	UN				
	U_2N_3				
	UN_2				
Thorium	Th_3N_4				
	ThN				
Beryllium	Be_3N_2	50			
Columbium	CbN				
	Cb_2N				
Vanadium	VN				
Silicon	Si_3N_4				Rockwell A99
Chromium	CrN				
	Cr_2N				
Aluminum	AlN				Mohs 7 ⁺ or 1200 kg/mm^2

TABLE 8
PHYSICAL PROPERTIES OF REFRACTORY BORIDES AT ROOM TEMPERATURES

Borides	Melting Temperature,	Boiling Temperature,		Density		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion, $F \times 10^6$	Coefficient of Thermal Conductivity,	
	C	F	C	F	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Hafnium									
HfB ₂	3250	5880			12.8	0.402			
Tantalum					10.5	0.380	3.06	6.3	0.026
TaB ₂	3200	5785			11.7	0.423			
Ta ₂ B ₅	2625	4750			13.5	0.488			
TaB ₃	2400	4350			14.0	0.506			
Zirconium					6.1	0.221	3.06	14.0	0.058
ZrB ₂	3040	5500			3.7	0.134		7.0	0.029
ZrB ₁₂	2680	4860			6.6	0.239		9.67	0.040
Columbium					7.5	0.271			
CbB ₂	2000	3650							
Cb ₃ B ₄					4.5	0.163		14.0	0.058
Titanium					16.7	0.603			
TiB ₂	2980	5400			16.0	0.578			
Tungsten					10.8	0.390			
W ₂ B ₅	2770	5020			8.8	0.318			
W ₂ B ₆	2860	5180			7.8	0.282			
Molybdenum					9.5	0.336			
MoB ₂	2180	3960			5.1	0.185			
MoB ₃	2100	3810			5.2	0.188			
Mo ₂ B ₃	2000	3650			5.6	0.203			
Vanadium					6.1	0.221			
VB ₂	2100	3810			8.45	0.224			
VB ₃					6.08	0.305			
Chromium									
CrB ₂	1850	3360							
Cr ₃ B ₂	1960	3560							
CrB ₃	1550	2820							
Thorium									
ThB ₄	2500	4530							
ThB ₆									

TABLE 9
MECHANICAL PROPERTIES OF REFRACTORY BORIDES
AT ROOM TEMPERATURES

Borides		Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness
Hafnium	HfB ₂				
Tantalum	TaB ₂ Ta ₃ B ₄ TaB				2615 KHN Knoops
Zirconium	ZrB ₂ ZrB ₁₂		29		Mohs 8 or 2200 kg./mm ² Rockwell A92-92.5
Columbium	CbB ₂ CbB Cb ₃ B ₄				Mohs 8+ 2600 kg./mm ²
Titanium	TiB ₂		19		Mohs 9+ 3400 kg./mm ²
Tungsten	W ₂ B WB W ₂ B ₅				2530 KHN Mohs 9
Molybdenum	MoB MoB ₂ Mo ₂ B				Mohs 8 1570 kg./mm ² 1280 kg./mm ² Mohs 8-9 1660 kg./mm ²
Vanadium	VB ₂ VB				Mohs 8-9
Chromium	CrB ₂ Cr ₃ B ₂ CrB				1800 kg./mm ² Mohs 9+ Mohs 8.5

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TABLE 10
PHYSICAL PROPERTIES OF REFRACTORY OXIDES AT ROOM TEMPERATURES

Oxides Melting Point Greater Than 1800 C	Melting Temperature,		Boiling Temperature,		Density		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion, F x 10 ⁶	Coefficient of Thermal Conductivity,	
	C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Thorium	3050	5520	4400	7950	9.69	0.350	0.007	8.0-9.0	4.84	0.02 0.024 at 100 C
Uranium	2880	5220			10.9	0.394				0.098
Magnesium	2800	5070	2825	5117	3.58	0.129	0.23	7.9	23.8	
Hafnium	2790	5060			9.6**	0.347	0.08	3.3		
Zirconium	2690	4870	4260	7700	6.1**	0.220	0.108	3.1	7.5	0.031 0.033 at 100 C
Calcium	2600	4710	2850	5162	3.32	0.120		7.55		
Cerium	2590	4700			7.1	0.257	0.090		4.9	0.024 0.025 at 100 C
Beryllium	2550	4620	4260	7700	3.0	0.108	0.03-0.05	5.28		
Strontium	2430	4410	3000	5432	4.7	0.170				
Yttrium	2410	4370	4300	7770	4.84	0.175				
Lanthanum	2300	4170	4200	7580	6.51	0.235				
Aluminum	2080	3772	3530	6380	3.97	0.143	0.21	4.2	16.2	0.067
Titanium	1800	3270	*	*	4.25**	0.154	0.16	4.2	3.8	0.016

* Decomposes at 3800 F

** Density depends on crystalline form

TABLE 10
Continued

Oxides Melting Point Greater Than 1800 C	Melting Temperature, °C		Boiling Temperature, °C		Density		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion $F \times 10^6$	Coefficient of Thermal Conductivity,	
	C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr-ft/F	Cal/cm/sec/C
Silicon	1723	3132	2950	5342	2.32**	0.084				
Zinc	1975	3587			5.66	0.204				
Barium	1917	3485	2200	3992	5.72	0.207				
Chromium	2265	4109	3000	5432	5.21	0.188				
Nickel	1950	3542			6.8	0.246				
Tantalum	1890	3434	3000	5432	8.02	0.290				
Vanadium	1977	3590			4.87	0.176				

* Decomposes at 3800 F

** Density depends on crystalline form

SiO₂ { (low quartz) 2.65 gm/cm³
 (low tridymite) 2.26 gm/cm³
 (low cristobalite) 2.32 gm/cm³
 (vitreous) 2.20 gm/cm³

TiO₂ { (brookite) 4.17 gm/cm³
 (rutile) 4.24 gm/cm³
 (anatase) 3.84 gm/cm³

ZrO₂ { (rhombic, cubic or trigonal) 6.27 gm/cm³
 (tetragonal) 6.10 gm/cm³
 (monoclinic) 5.56 gm/cm³

HfO₂ { (monoclinic) 9.68 gm/cm³
 (tetragonal) 10.01 gm/cm³

TABLE 11
MECHANICAL PROPERTIES OF REFRACTORY OXIDES
AT ROOM TEMPERATURES

Oxides Melting Point Greater Than 1800 C		Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Mohs Hardness
Thorium	ThO_2	18-20	14		6.7
Uranium	UO_2				
Magnesium	MgO	28	13		5.9
Hafnium	HfO_2				
Zirconium	ZrO_2	27	22		6.9
Calcium	CaO				4.5
Cerium	CeO_2				6.0
Beryllium	BeO	45	14		9.0
Strontium	SrO				3.5
Yttrium	Y_2O_3				
Lanthanum	La_2O_3				
Aluminum	Al_2O_3	50	37		9.0
Titanium	TiO_2	14	8		5.9
Silicon	SiO_2	8.5	15.6		6.5
Zinc	ZnO				4 to 4.5
	ZnO				6.5
Barium	BaO				3.3
Nickel	NiO				5.5

TABLE 12
PHYSICAL PROPERTIES OF REFRACTORY SILICIDES AT ROOM TEMPERATURES

Silicides		Melting Temperature,		Boiling Temperature,		Density		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion, $F \times 10^6$	Coefficient of Thermal Conductivity,	
		C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Tantalum	TaSi ₂	2400	4350			8.8	0.318				
Tungsten	WSi ₂	2150	3900			9.8	0.354				
Titanium	Ti ₂ Si ₃	2120	3850			4.32	0.156				
	TiSi ₂	1540	2800			4.13	0.149				
Molybdenum	Mo ₃ Si ₂	2100	3780			7.4	0.268				
	Mo ₂ Si	2050	3690			8.97	0.324				
Columbium	MoSi ₂	2030	3650			6.1	0.220				
	CoSi ₂	1950	3540			5.29	0.191				
Vanadium	VSi ₂	1750	3180			4.7	0.170				
Chromium	Cr ₃ Si					6.52	0.236				
	CrSi ₂	1570	2860			4.4	0.159				
Zirconium	ZrSi ₂	1520	2770			4.9	0.177				

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TABLE 13
MECHANICAL PROPERTIES OF REFRACTORY SILICIDES
AT ROOM TEMPERATURES

Silicides		Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	kg./mm^2 Hardness
Tantalum	TaSi ₂	59			1200
Tungsten	WSi ₂				1090
Titanium	Ti ₅ Si ₃				986
	TiSi ₂				870
Molybdenum	Mo ₃ Si ₂				1170
	Mo ₃ Si				1310
	MoSi ₂				1290
Columbium	CbSi ₂				1050
Vanadium	VSi ₂				
Chromium	Cr ₃ Si				
	CrSi ₂				1150
Zirconium	ZrSi ₂				1030

TABLE 14
PHYSICAL PROPERTIES OF REFRACTORY SULFIDES
AT 1800° TEMPERATURES

Sulfides	Melting Temperature,		Boiling Temperature,		Density,		Specific Heat, Btu/lb/°F or Cal/gm/°C	Coefficient of Thermal Expansion F x 10 ⁶	Coefficient of Thermal Conductivity	
	C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Cerium	2450	4442			5.93	0.214		13.2		
Thorium	Ce ₃ S ₄	2050	3722		5.3	0.191				
	ThS	2200	3992		9.57	0.346				
	Th ₄ S ₇	2000	3632		7.78	0.281				
	Th ₂ S ₃	1950	3542		7.88	0.285				
Barium	ThS ₂	1905	3461		7.36	0.266				
	BaS	2200	3992		4.3	0.155				
	Nd ₂ S ₃	2200	3992		5.34	0.193				
Neodymium										
Hafnium	HfS	2100	3812							
		2200	3992							
Lanthanum	La ₂ S ₃	2100	3812		5.00	0.181				
Zirconium	Zr ₄ S ₃	2150	3902							
Titanium		2050	3722							
	TiS	2100	3812							
Uranium	US	2000	3632		4.05	0.146				
	U ₂ S ₃	2010	3650							
Strontium	US ₂	2000	3632		10.87	0.393				
	SrS	1850	3362		2.36	0.085				
Magnesium	MgS	2000	3692		7.90	0.285				
		2000	3692		3.65	0.132				
Yttrium	Y ₂ S ₃	1900	3452		2.85	0.103				
Samarium		1950	3542							
	Sm ₂ S ₃	1900	3452		5.73	0.207				
Vanadium	VS	1800	3272		4.4	0.159				
		2000	3692							

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TABLE 15
PHYSICAL PROPERTIES OF INTERMETALLIC REFRACTORY
MATERIALS AT ROOM TEMPERATURES

Inter-Metallic		Melting Temperature,		Boiling Temperature,		Density,		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion F x 10 ⁶	Coefficient of Thermal Conductivity	
		C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Tantalum	90% -10 W	3031	5497			16.9	0.608				
Molybdenum Tungsten	70 M- 30 W 85 M- 15 W		5145			11.9	0.43				

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TABLE 16
MECHANICAL PROPERTIES OF INTERMETALLIC REFRACTORY
MATERIALS AT ROOM TEMPERATURES

Intermetallic	Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness	
Tantalum Tungsten	30	142			90-percent cold-worked sheet
		80			Fully annealed and re- crystallized
Molybdenum Tungsten	50	121	106		Stress relieved 2000 F
		65.7			Same as above with test temperature 1800 F
		83.8	69.1		Recrystallized 2600 F
		32.4			Same as above but test temperature 1800 F

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TABLE 17
PHYSICAL PROPERTIES OF SOME NONMETALLIC
REFRACTORIES AT ROOM TEMPERATURE

	Melting Point,		Boiling Point,		Density,		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion F x 10 ⁶	Coefficient of Thermal Conductivity	
	C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Nonmetallic										
Boron	2050	3715	2750	4620	2.3	0.083				
Silicon										
Crystalline	1420	2592	2600	4710	2.4	0.087				
Graphitic			2600	4710	2-2.5	0.072-0.090				
Amorphous Carbon			2600	4710	2.0	0.072				
Amorphous										
	Sublimation		4200	7592	1.8-2.1	0.065-0.076				
Diamond	3649	6600	4200	7592	3.51	0.127				
Graphite (1)	3649	6600	4200	7592	1.6-2	0.058-0.072	0.17	1.6	92	0.380
Pyrolytic Graphite (2)	3649	6600	4200	7592	2.2	0.079	0.23	a=-0.70 c=7.2	a=234 c=0.54 a=155 c=0.40	a=0.967 c=0.002 a=0.641 c=0.002
Boron Pyralloy					2.22	0.080				

(1) Stackpole

(2) High Temperature Material -

"a" Direction is parallel to surface of deposition and hence the basal planes
"c" Direction is normal to surface and basal planes

TABLE 17

Continued

	Melting Point,		Boiling Point,		Density, lb/in. ³		Specific Heat, Btu/lb/F or Cal/gm/C	Coefficient of Thermal Expansion F x 10 ⁶	Coefficient of Thermal Conductivity	
	C	F	C	F	gm/cm ³	lb/in. ³			Btu/hr/ft/F	Cal/cm/sec/C
Nonmetallic										
ATJ(3)	3649	6600	4200	7592	1.73	0.062		a=1.28 c=1.94	a=65.3 c=53.2	a=0.27 c=0.22
ZTA(3)	3649	6600	4200	7592	1.95	0.070		a=0.44 c=4.00	a=94.3 c=43.5	a=0.39 c=0.18
AHIG-1A(5)	3649	(6) 6600			1.9-	0.069-	0.174		100-120	0.413-
AHIG-2(5)	3760	(6) 6800			1.985	0.072	0.208-		170	0.496
Clear Fused Quartz(h)	(Softening Point) 1667	3032			2.05-	0.074-	0.241			0.703
Type F Glass(h)	(Softening Point) 915	1679			2.10	0.076		0.30 0 to 572 F	0.798	0.0033
					2.2	0.079	0.165	2.33 0 to 572 F		
					2.53	0.091	0.170			

(3) National Carbon Co., typical values

(4) General Electric

(5) American Metal Products Co.

(6) Volatility Point (melts at 8600 F under pressure)

"a" Direction is parallel to surface of deposition and hence the basal planes

"b" Direction is normal to surface and basal planes

TABLE 18
MECHANICAL PROPERTIES OF SOME NONMETALLIC
REFRACTORIES AT ROOM TEMPERATURE

Nonmetallic	Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness	
Boron	50				
Silicon					
Crystalline					
Graphitic					
Amorphous					
Carbon					
Amorphous					
Diamond					
Graphite (1)	0.67 to 1.4	2 to 4		Mohs 0.5 to 1	Shear Strength, psi a=1400 c=5300
Pyrolytic	a=4.25 in tension	a=13000 c= 4000	Bend Strength, psi 22.7	Mohs a=1 c=4.5	Compressive Strength a=15,000 c=60,000
Graphite (2)	a=3.5 in compression				Shear 1200
Boron	5.8	14,700			
Pyralloy					

(1) Stackpole
(2) High Temperature Materials

"a" Direction is parallel to surface of deposition and hence the basal planes
"c" Direction is normal to surface and basal planes

TABLE 18
Continued

Nonmetallic	Modulus of Elasticity $\times 10^{-6}$	Ultimate Tensile Strength, $\text{psi} \times 10^{-3}$	Yield Strength, $\text{psi} \times 10^{-3}$	Hardness	
ATJ (3)	a=1.40 c=1.15		Flexural a=3.32 c=3.33		Compressive a=8380 c=8580
ZTA (3)	a=2.40 c=0.90		Flexural a=5.50 c=2.90		Compressive a=12,500 c=13,000
AHDG-1A (5)	1.35	4 to 6	Flexural a=5 to 6 c=0.85 a		
AHDG-2 (5)	1.5	5 to 8	Flexural 8 esti- mated		Compressed 15,000 to 30,000 (estimated)
Clear Fused Quartz (4)	10				
Type E Glass (4)	12.7				

(3) National Carbon Co., typical values
 (4) General Electric
 (5) American Metal Products Co.

"a" Direction is parallel to surface of
 deposition and hence the basal planes
 "c" Direction is normal to surface and
 basal planes

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TABLE 19

REFRACTORY METAL PRODUCERS

Primary Producers	Metal												
	Tungsten	Molybdenum	Tantalum	Columbium	Chromium	Vanadium	Niobium	Platinum	Palladium	Osmium	Ruthenium	Rhodium	Iridium
ANACONDA CO., New York								X	X				
BISHOP, J., & CO., Malvern, Pa.								X	X	X	X	X	X
CARBORUNDUM METALS CO., Akron, N. Y., Div. Carborundum Co.						X							
CHASE BRASS & COPPER CO., Waterbury, Conn.													X
CLEVELAND TUNGSTEN INC., Cleveland, sub. Molybdenum Corp. of America	X												
CLIMAX MOLYBDENUM CO. OF MICHIGAN INC., Detroit, div. American Metal Climax		X											
DU PONT DE NEMOURS & CO. INC., E. I., Wilmington, Del.				X									
ELMET DIV., Lewiston, Maine, North American Philips Co. Inc.	X	X											
ENGELHARD INDUSTRIES INC., Newark, N. J.								X	X	X	X	X	X
FANSTEEL METALLURGICAL CORP., North Chicago, Ill.	X	X	X	X									
FIRTH STERLING INC., Pittsburgh	X												
FOOTE MINERAL CO., Philadelphia							X						
GENERAL ELECTRIC CO., Schenectady, N. Y.	X	X		X									
GOLDSMITH BROS., Chicago, div. National Lead Co.								X	X	X	X	X	X
INTERNATIONAL NICKEL CO., New York								X	X		X	X	X
JOHNSON-MATTHEY & CO. LTD., London								X	X	X	X	X	X
KAWECKI CHEMICAL CO. INC., New York			X	X									
KENAMETAL INC., Lafayette, Pa.	X		X	X									
METALS & CONTROLS DIV., Attleboro, Mass., Texas Instruments Inc.								X					
METALS & RESIDUES INC., Springfield, N. J.	X	X											
MOLYBDENUM CORP. OF AMERICA, Pittsburgh		X											
NATIONAL RESEARCH CORP., METALS DIV., Cambridge, Mass.			X										
OREGON METALLURGICAL CORP., Albany, Oreg.	X	X				X							
PHELPS DODGE CORP., New York								X	X				
REACTIVE METALS INC., Niles, Ohio			X	X			X						
REDUCTION & REFINING CO., Kenilworth, N. J.	X	X											
SHIELDALLOY CORP., Newfield, N. J.					X								
STAUFFER CHEMICAL CO., METALS DIV., Richmond, Calif.	X	X	X	X									
SYLVANIA ELECTRIC PRODUCTS INC., Chemical & Metallurgical Div., Tawanda, Pa.	X	X											
TEMESCAL METALLURGICAL CORP., Berkeley, Calif.		X	X	X									
UNION CARBIDE METALS CO., div. of Union Carbide Corp., New York	X		X	X	X	X							
UNIVERSAL-CYCLOPS STEEL CORP., Bridgeville, Pa.	X	X											
UNIVERSITY OF TENNESSEE, Department of Chemistry, Knoxville, Tenn.													X
VANADIUM CORP. OF AMERICA, New York						X							
WAN CHANG CORP., New York	X	X	X	X			X						
WESTINGHOUSE ELECTRIC CORP., Pittsburgh		X											

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TABLE 20
ROOM TEMPERATURE PROPERTIES OF TUNGSTEN

	Arc-Sprayed	Arc-Sprayed and Sintered	Cold-Pressed and Sintered
Percent of Theoretical Density	86 90*	93	93
Microstructure	Lamellar	Equiaxed Fine Grain	Equiaxed Fine Grain
Hardness, DPH	250	300	300
Ultimate Tensile Strength, psi (ring crush test)	22,000 30,000*	70,000	60,000
Forgeability	Poor	Good	Good

Data reported by Allison Division, General Motors Corporation

*Reported by Linde, Division, Union Carbide Company

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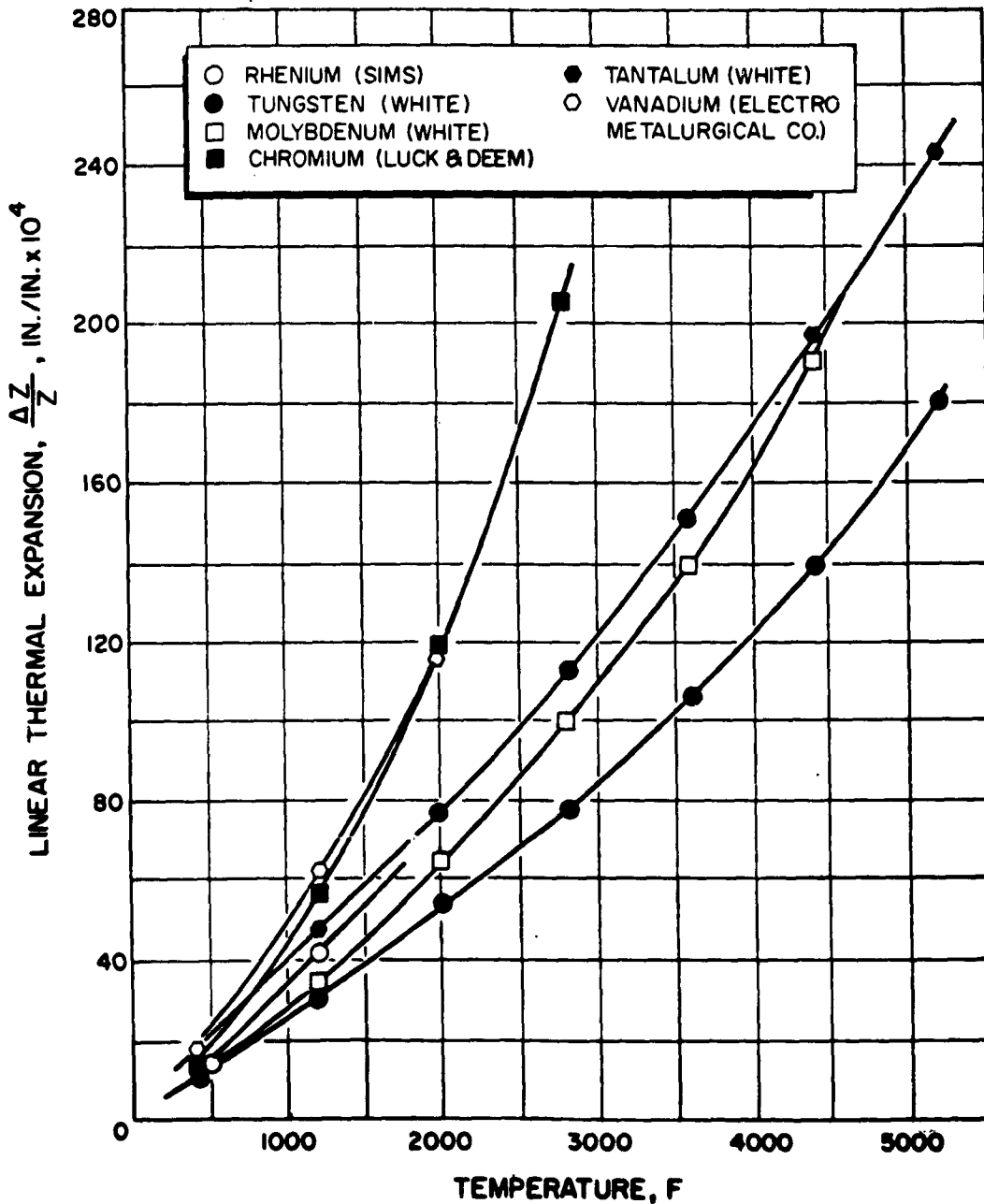


Figure 1. Thermal Expansion of Various Refractory Metals as a Function of Temperature

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B-3350-3

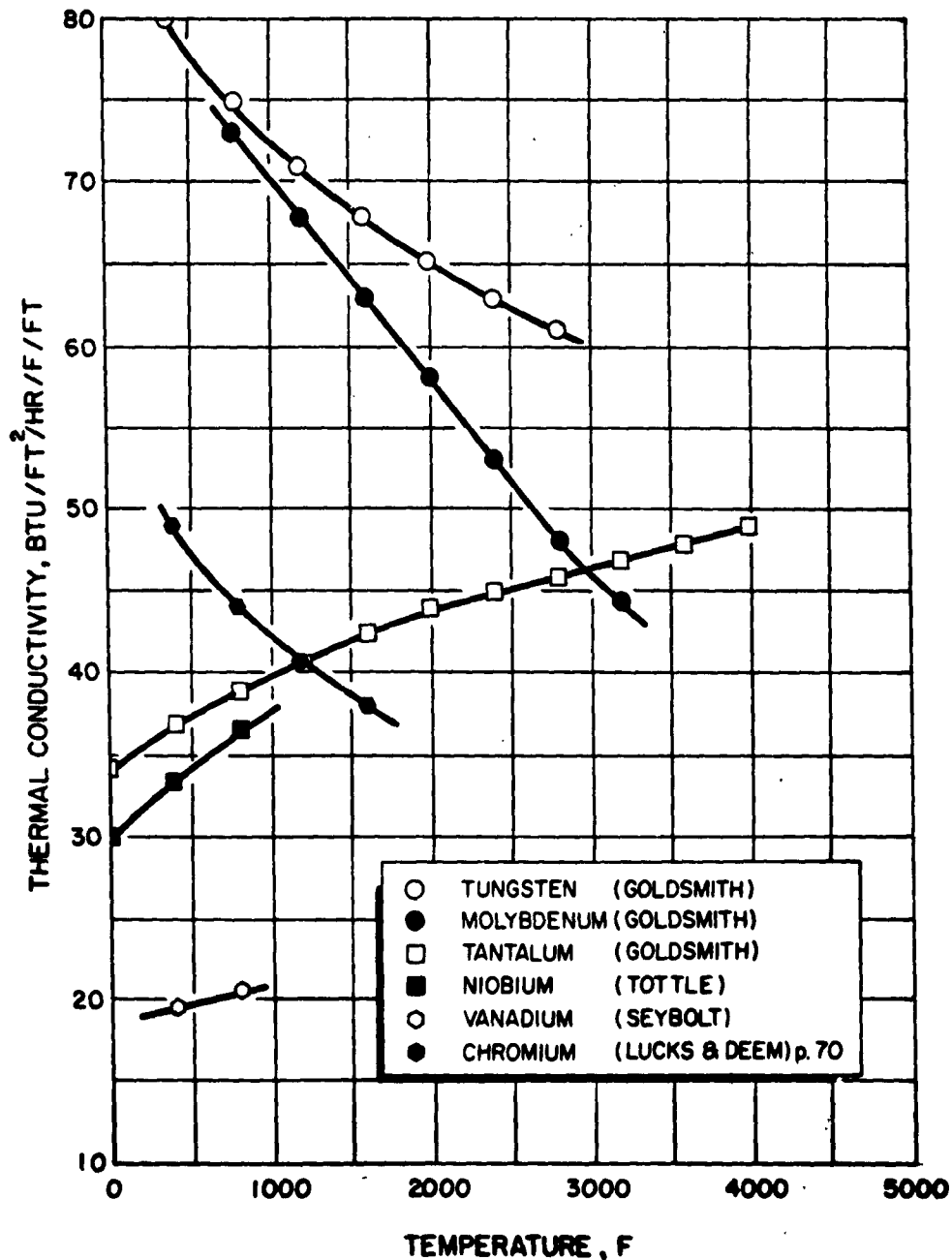


Figure 2 . Thermal Conductivity of Various Refractory Metals as a Function of Temperature

B-3350-3

III-117

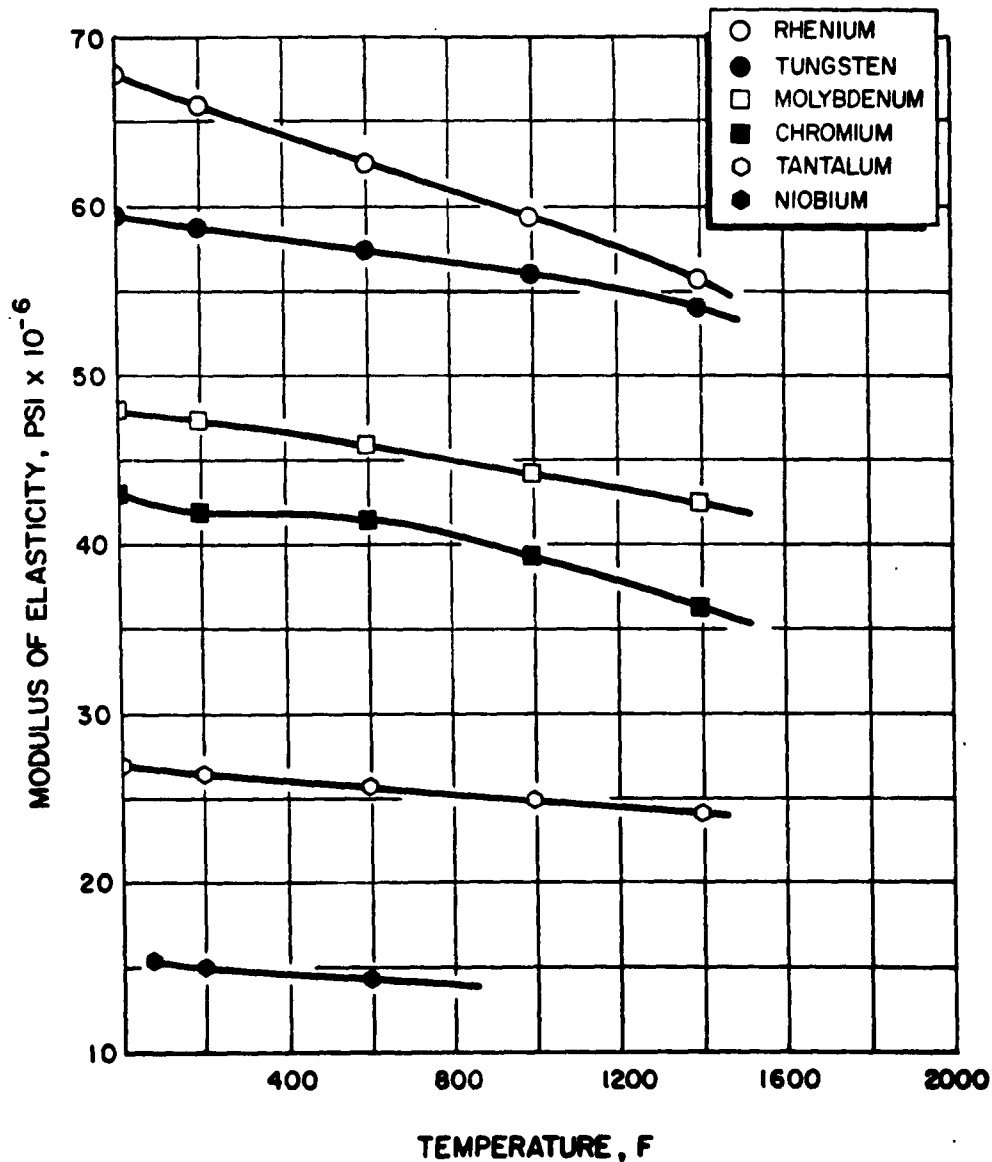


Figure 3 . Modulus of Elasticity as a Function of Test Temperature for Different Refractory Metals

III-118

B-5390-3

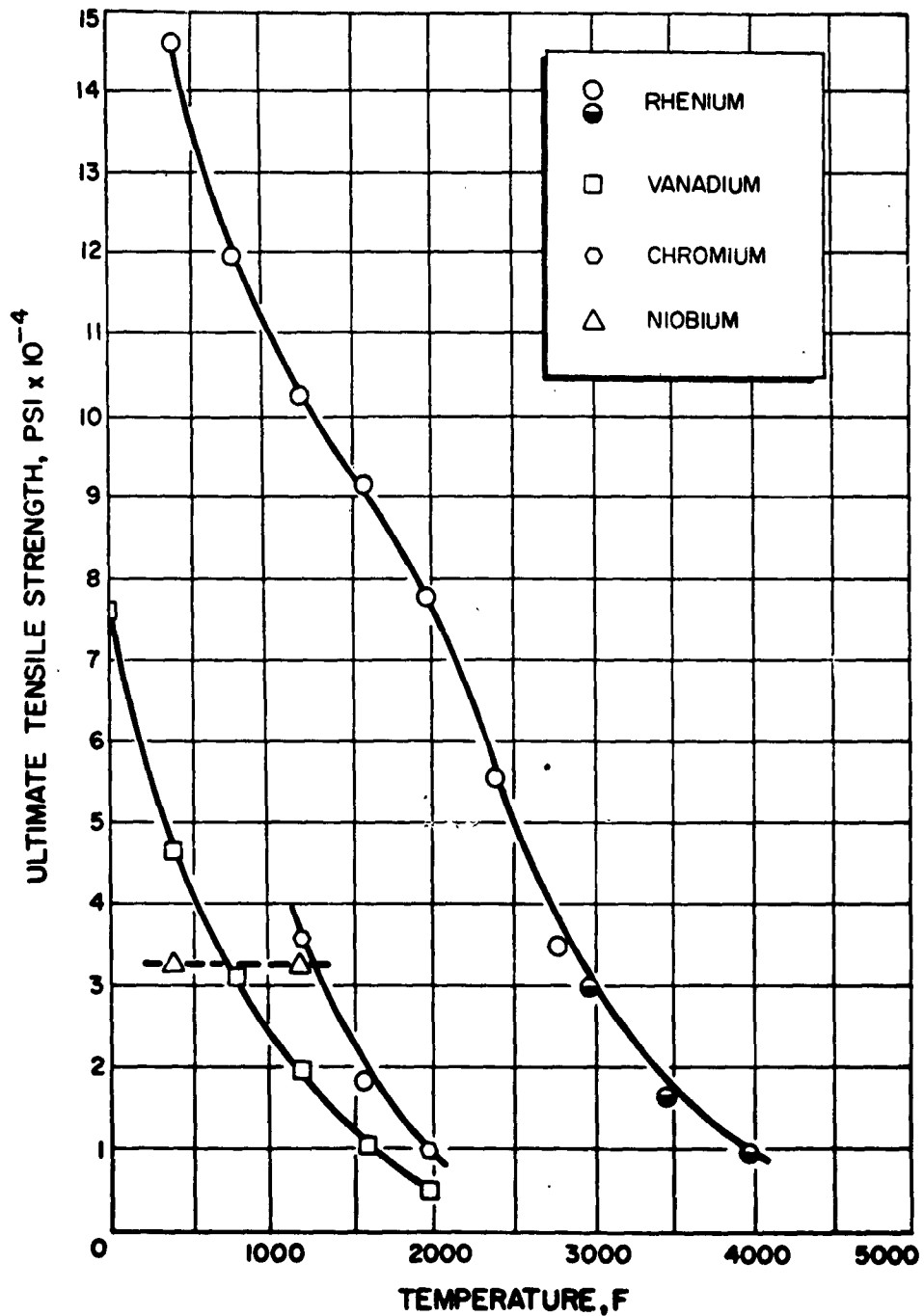


Figure 4. Effect of Temperature on the Tensile Strength of Rhenium, Niobium, Vanadium, and Chromium

R-3350-3

III-119

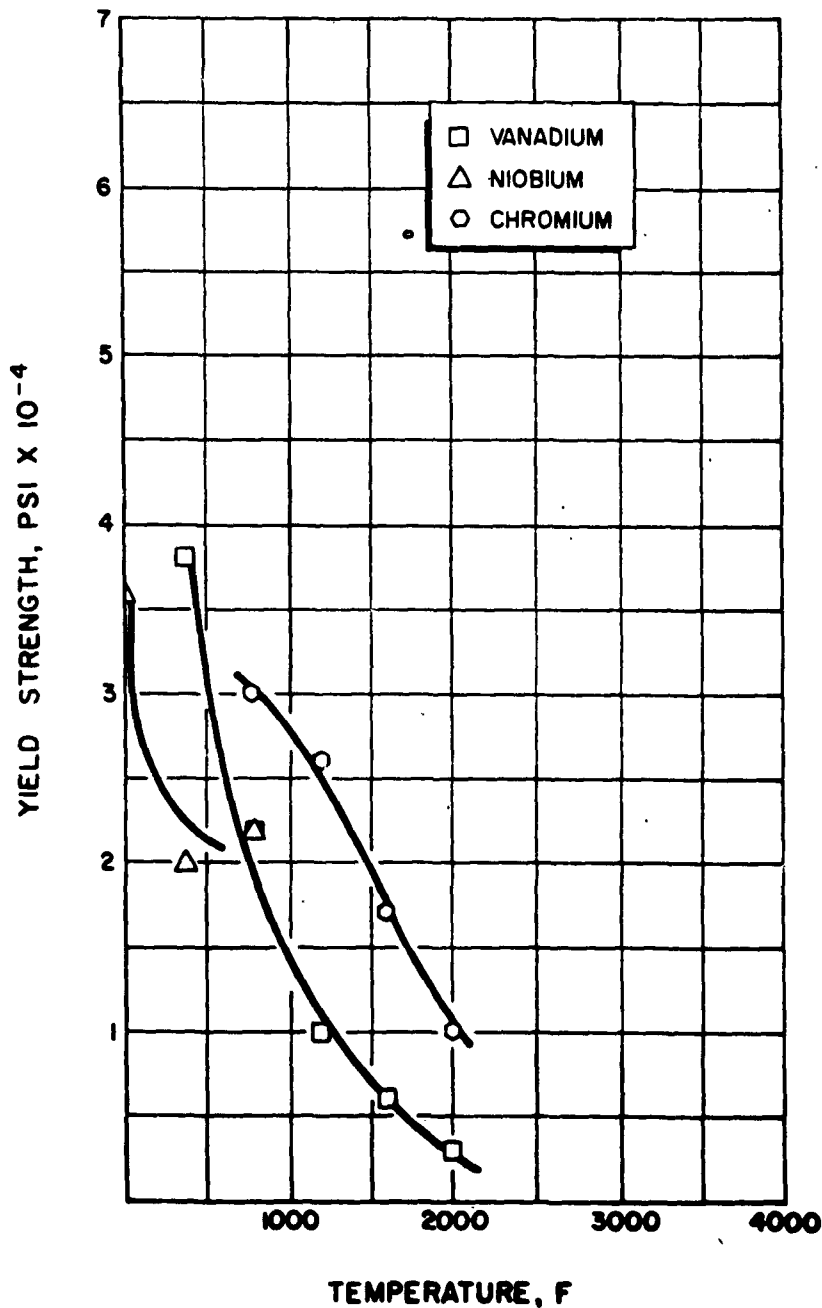


Figure 5 . Effect of Temperature on the Yield Strength of Chromium, Niobium, and Vanadium

III-120

B-3330-3

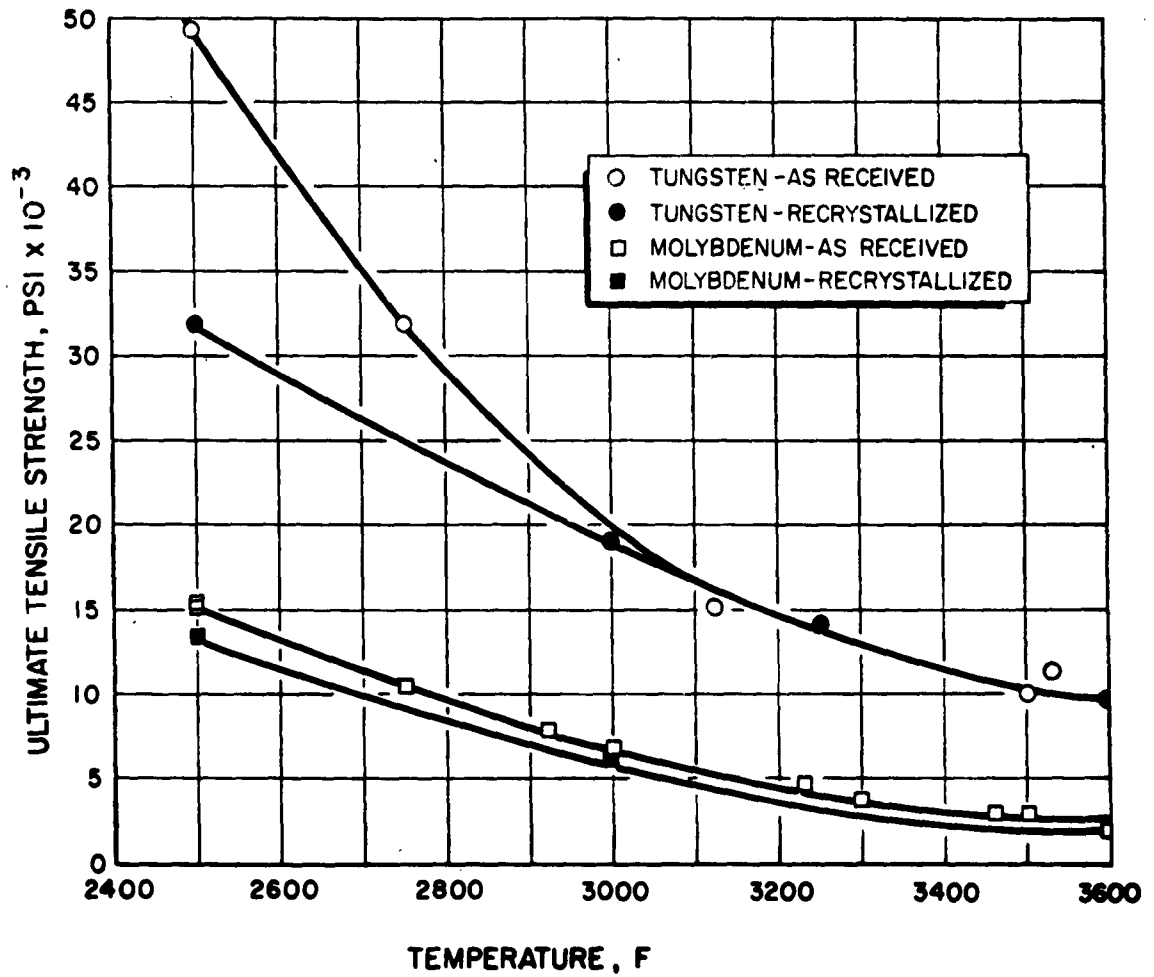


Figure 6 . Comparison of High-Temperature Tensile Strengths of Tungsten and Molybdenum Materials in the As Received and Recrystallized Condition

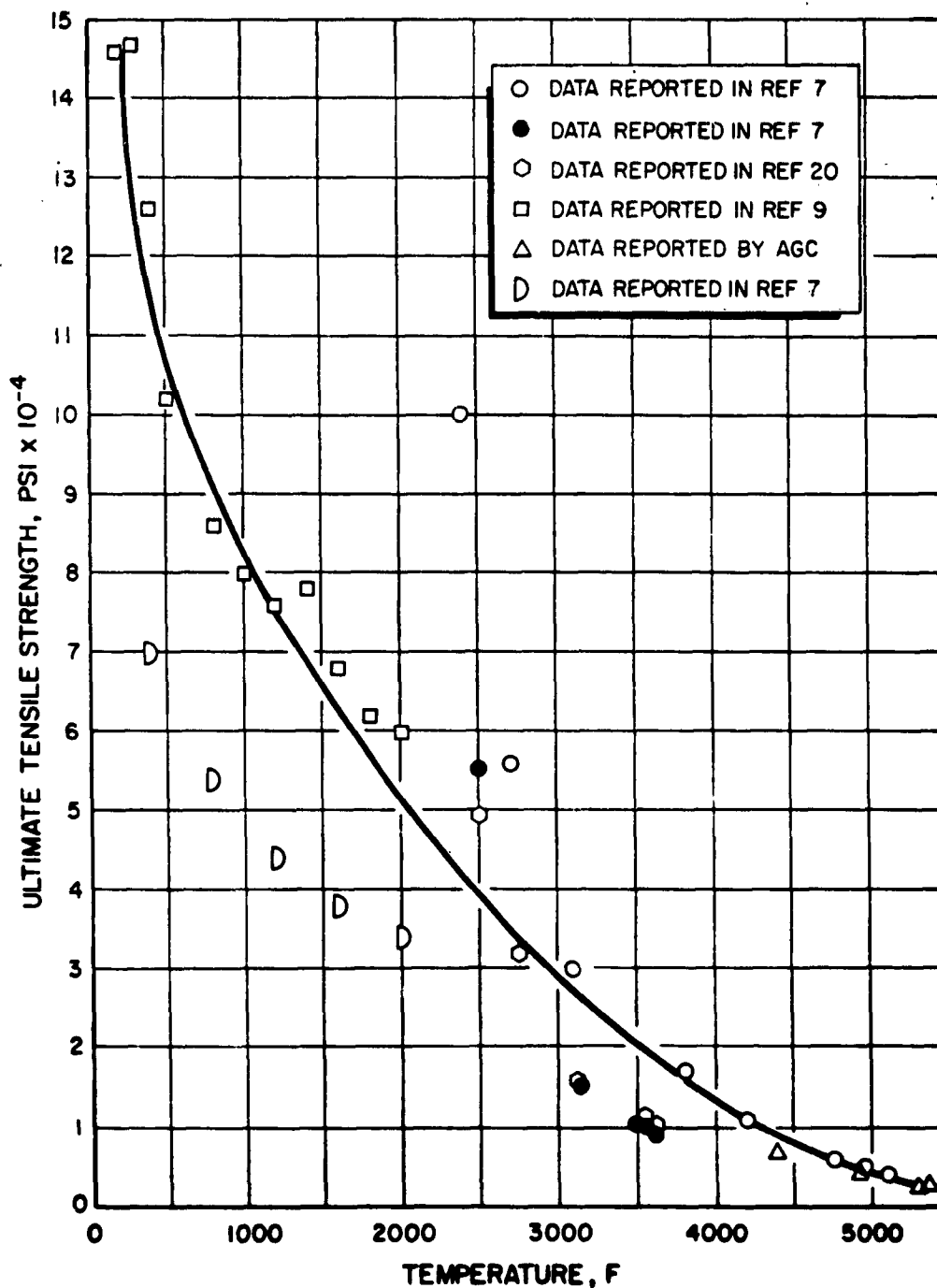


Figure 7. Effect of Temperature on the Tensile Strength of Wrought Tungsten

III-122

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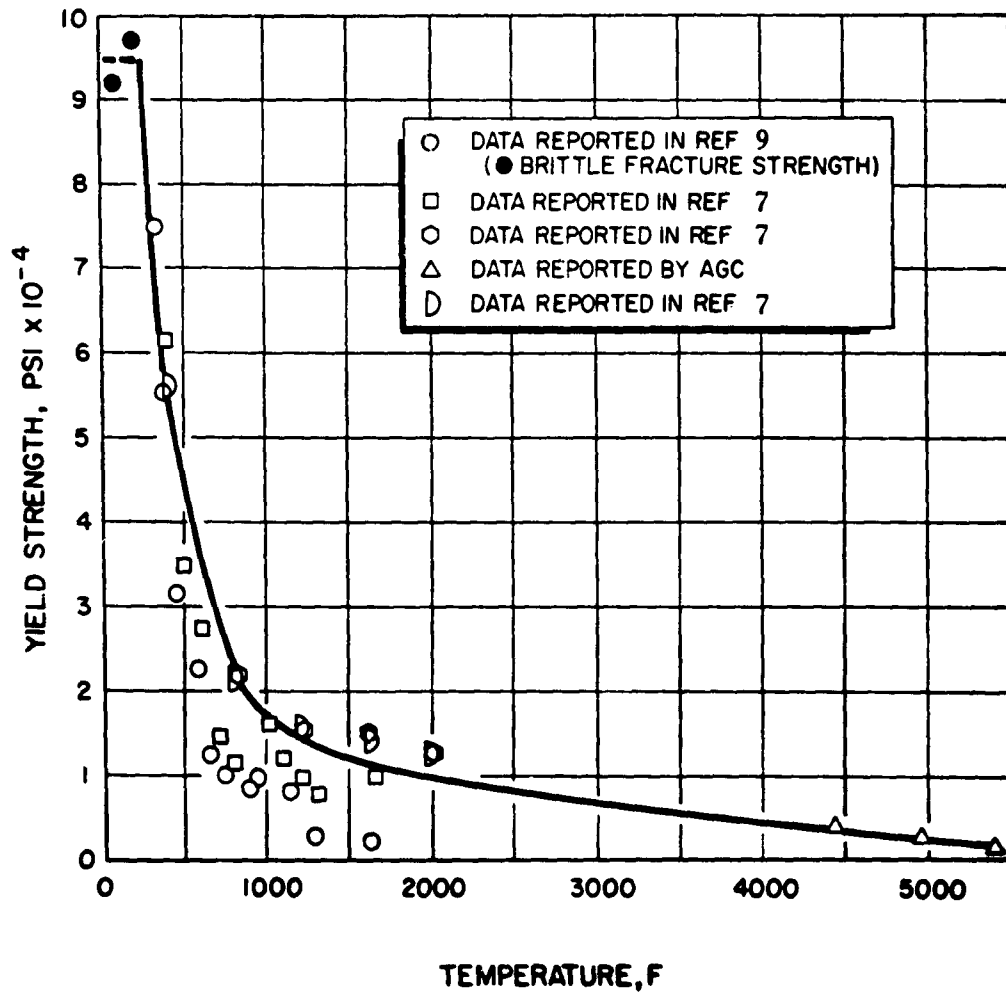


Figure 8. Effect of Temperature on the Yield Strength of Tungsten

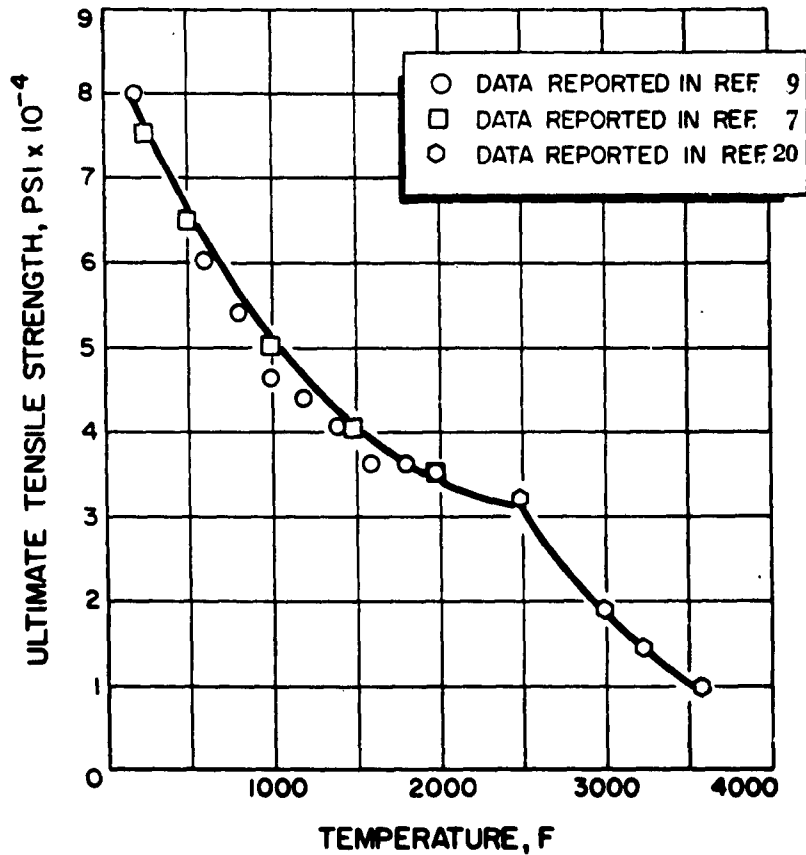


Figure 9. Effect of Temperature on Short Time Tensile Strength of Recrystallized Tungsten

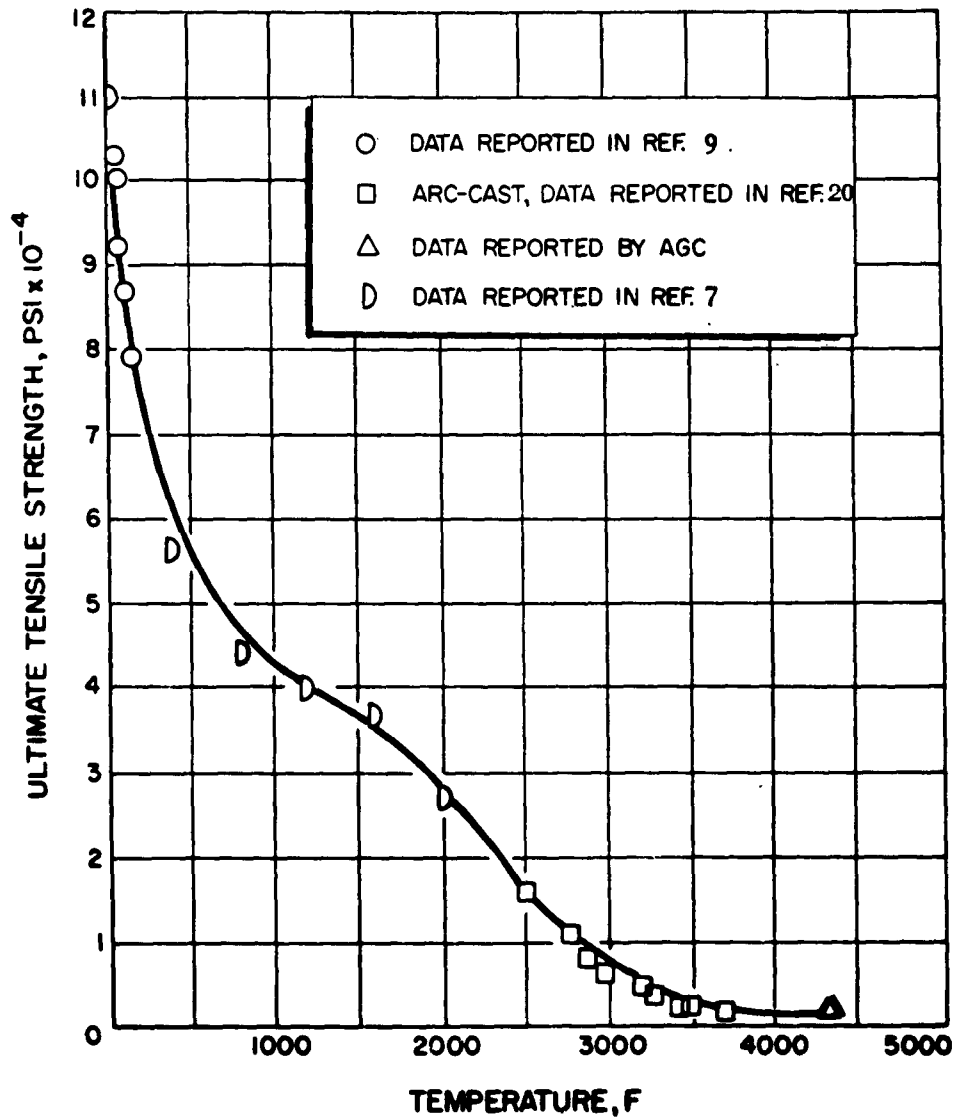


Figure 10. Effect of Temperature on the Tensile Strength of Wrought Molybdenum

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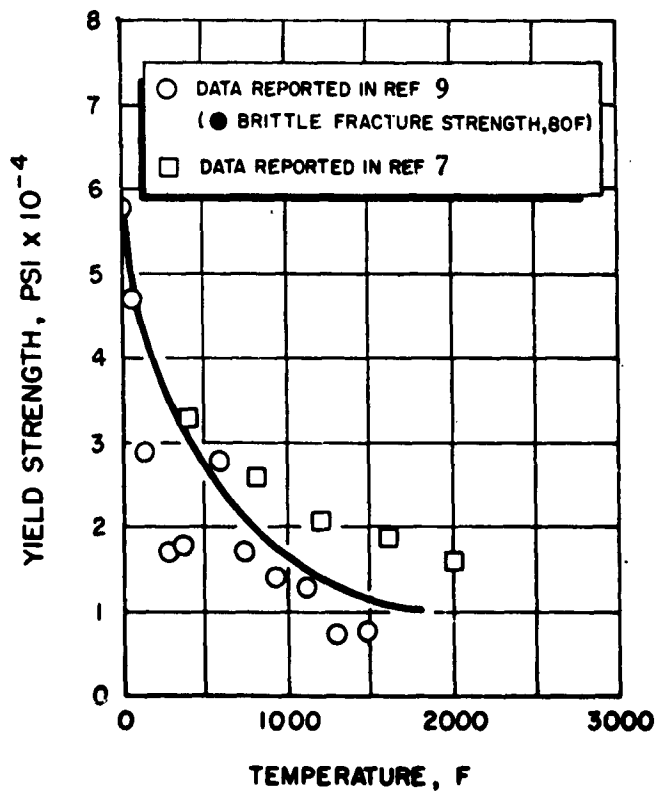


Figure 11. Effect of Temperature
on the Yield Strength
of Molybdenum

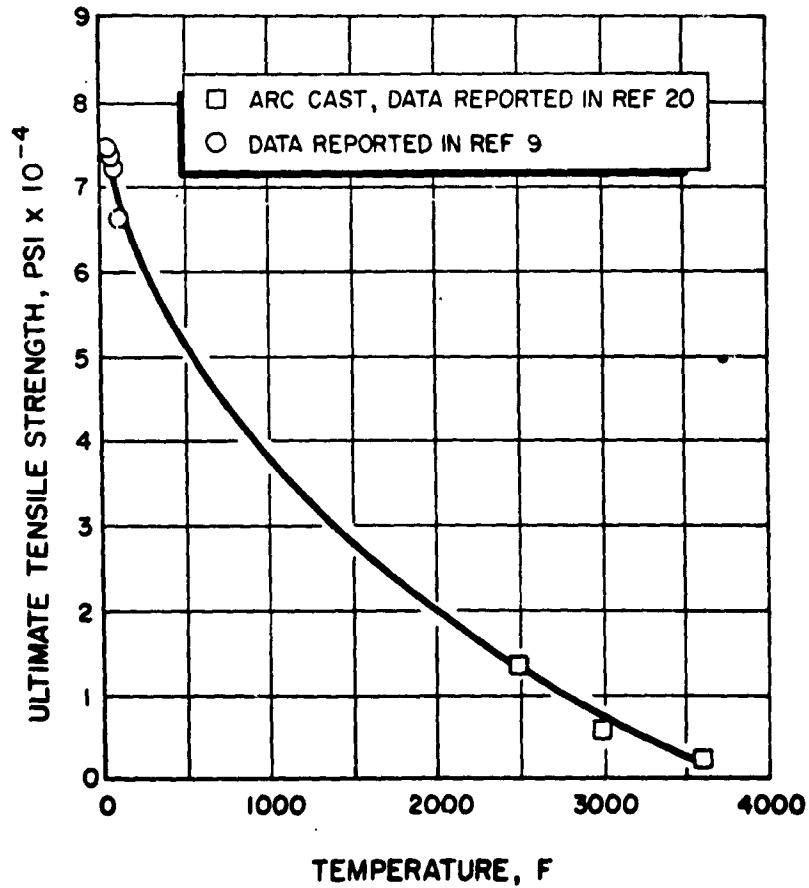


Figure 12. Effect of Temperature on the Tensile Strength of Recrystallized Molybdenum

B-3350-3

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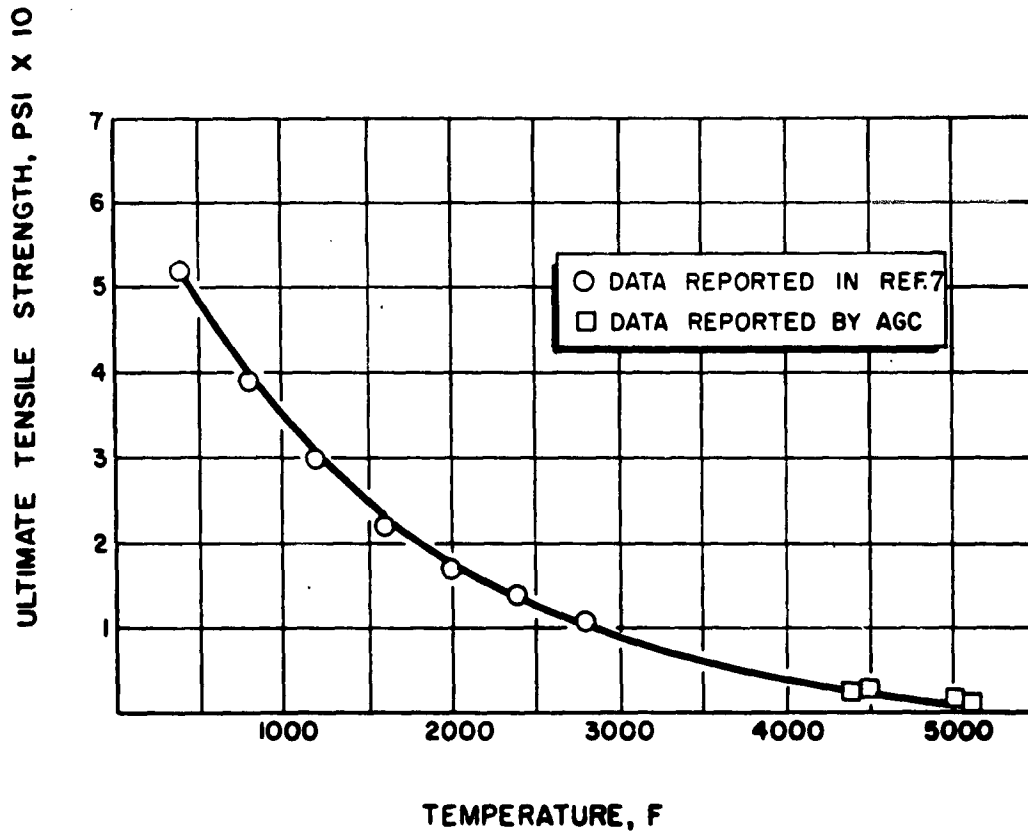


Figure 13. Effect of Temperature on the Tensile Strength of Tantalum

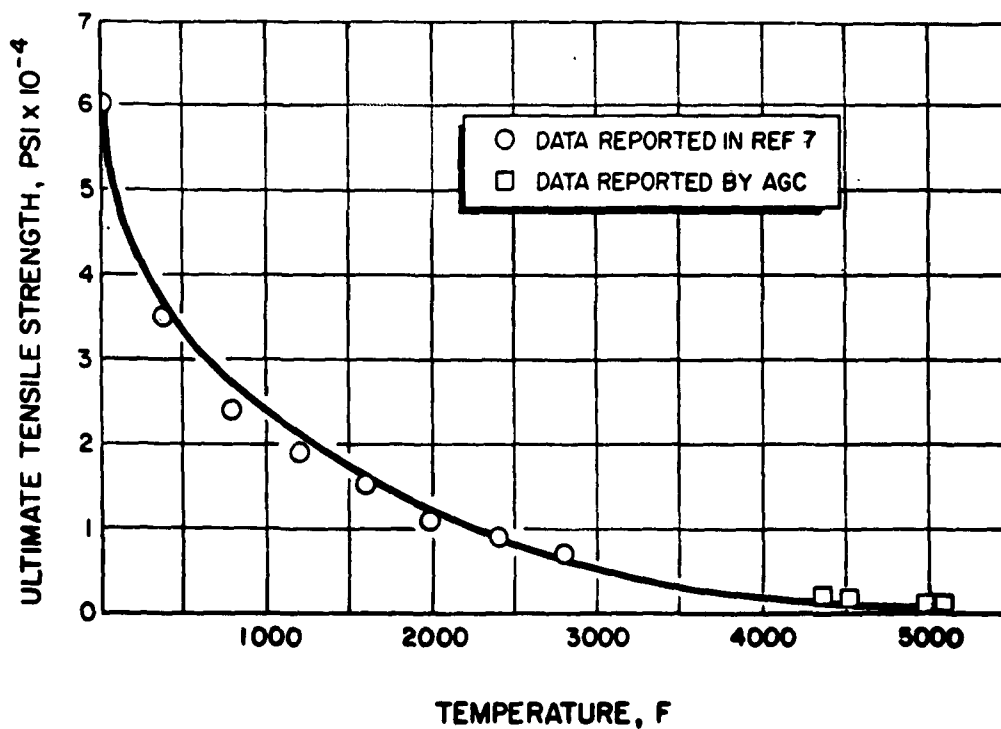


Figure 14. Effect of Temperature on the Yield Strength of Tantalum

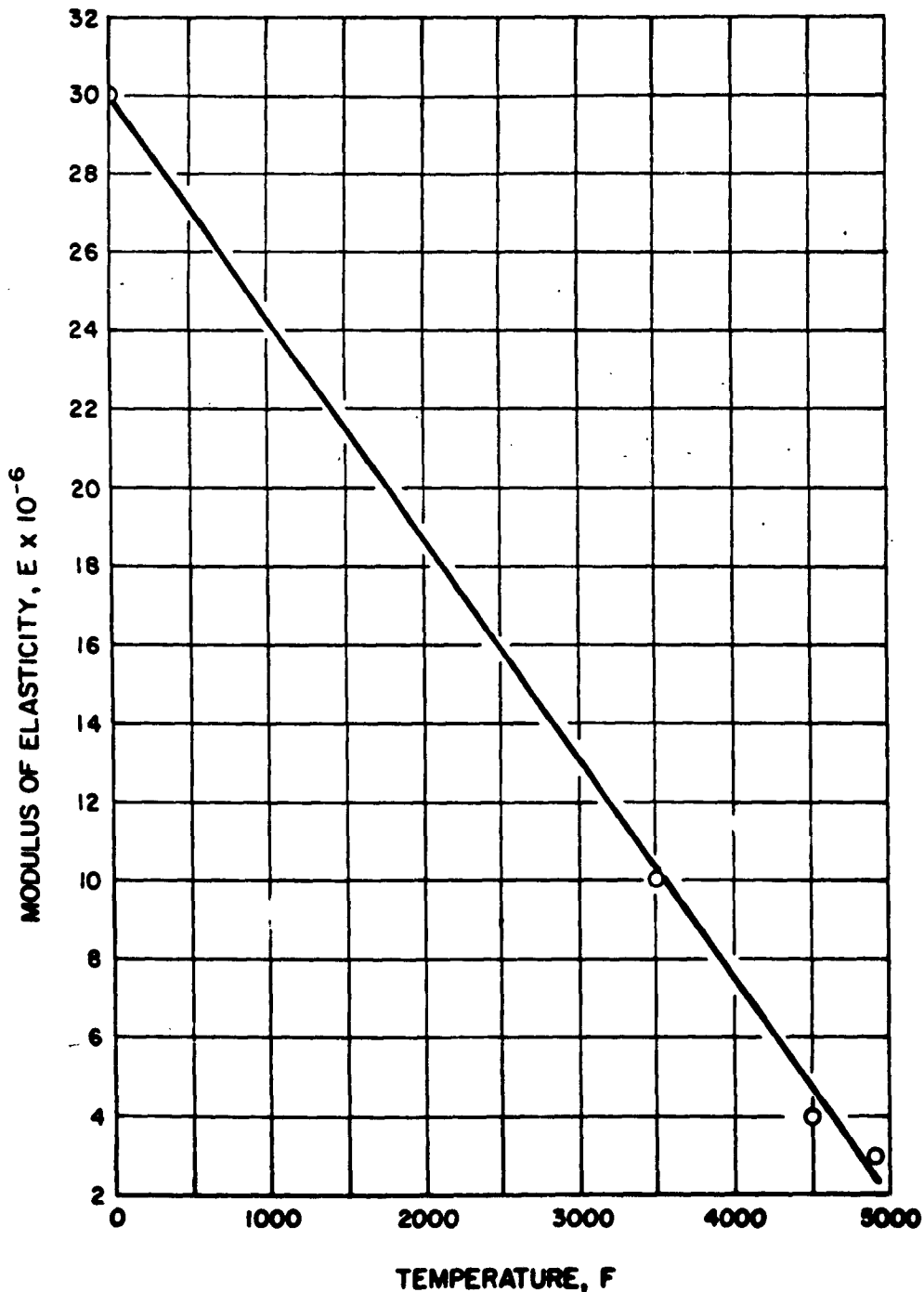


Figure 15 . Effect of Temperature on the Modulus of Elasticity of 90 Tantalum-10 Tungsten Alloy

III-130

B-3350-3

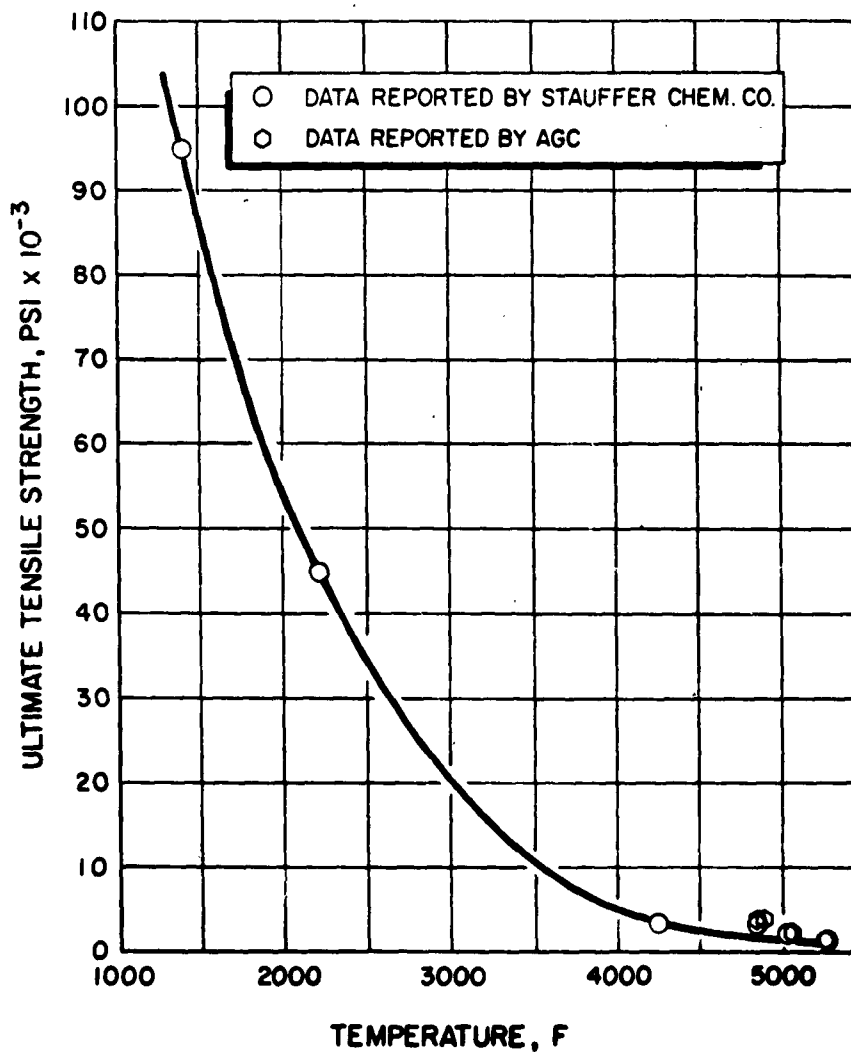


Figure 16 . Effect of Temperature on the Tensile Strength of 90 Tantalum-10 Tungsten Alloy

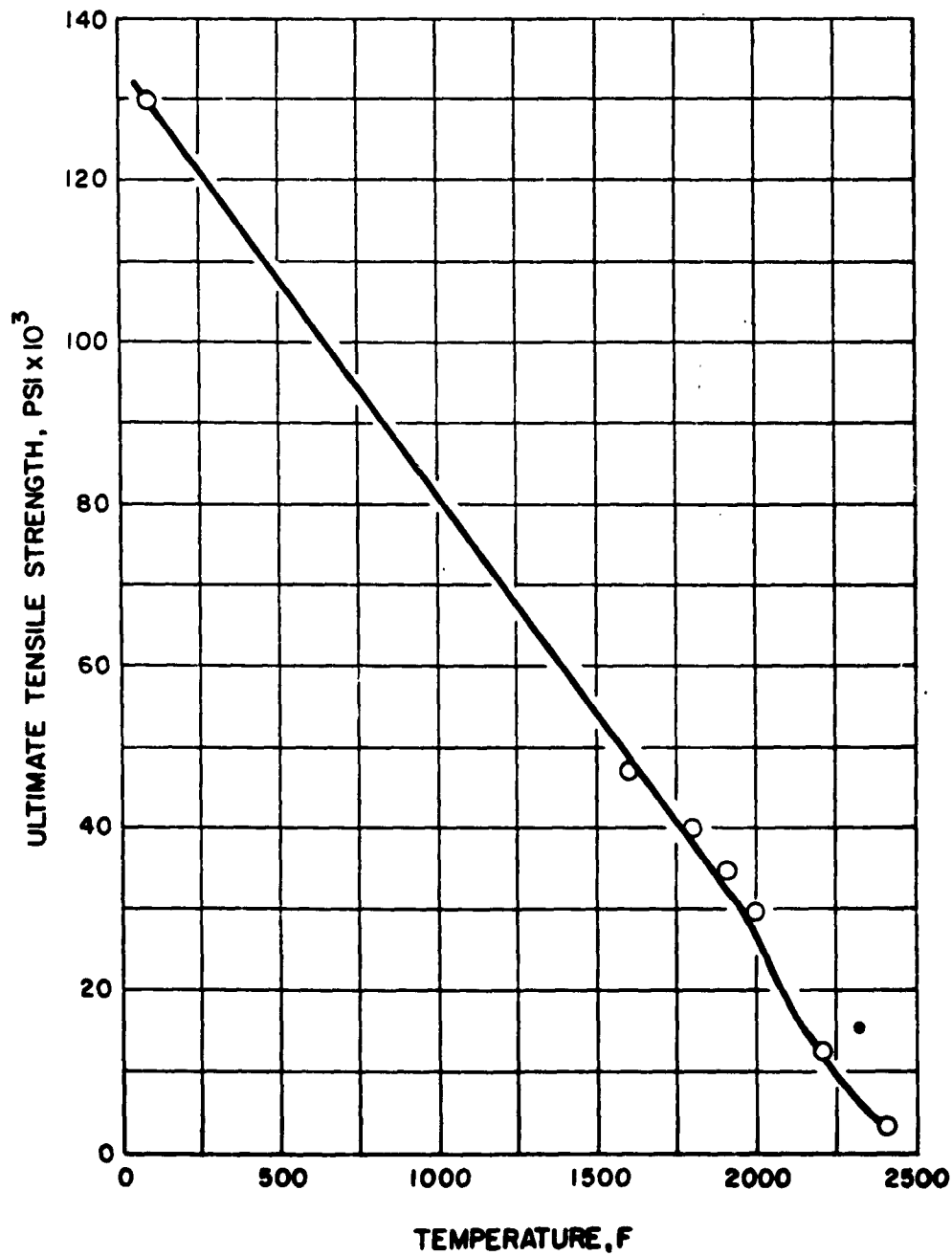


Figure 17. Effect of Temperature on the Tensile Properties of Titanium Carbide

III-132

B-3350-3

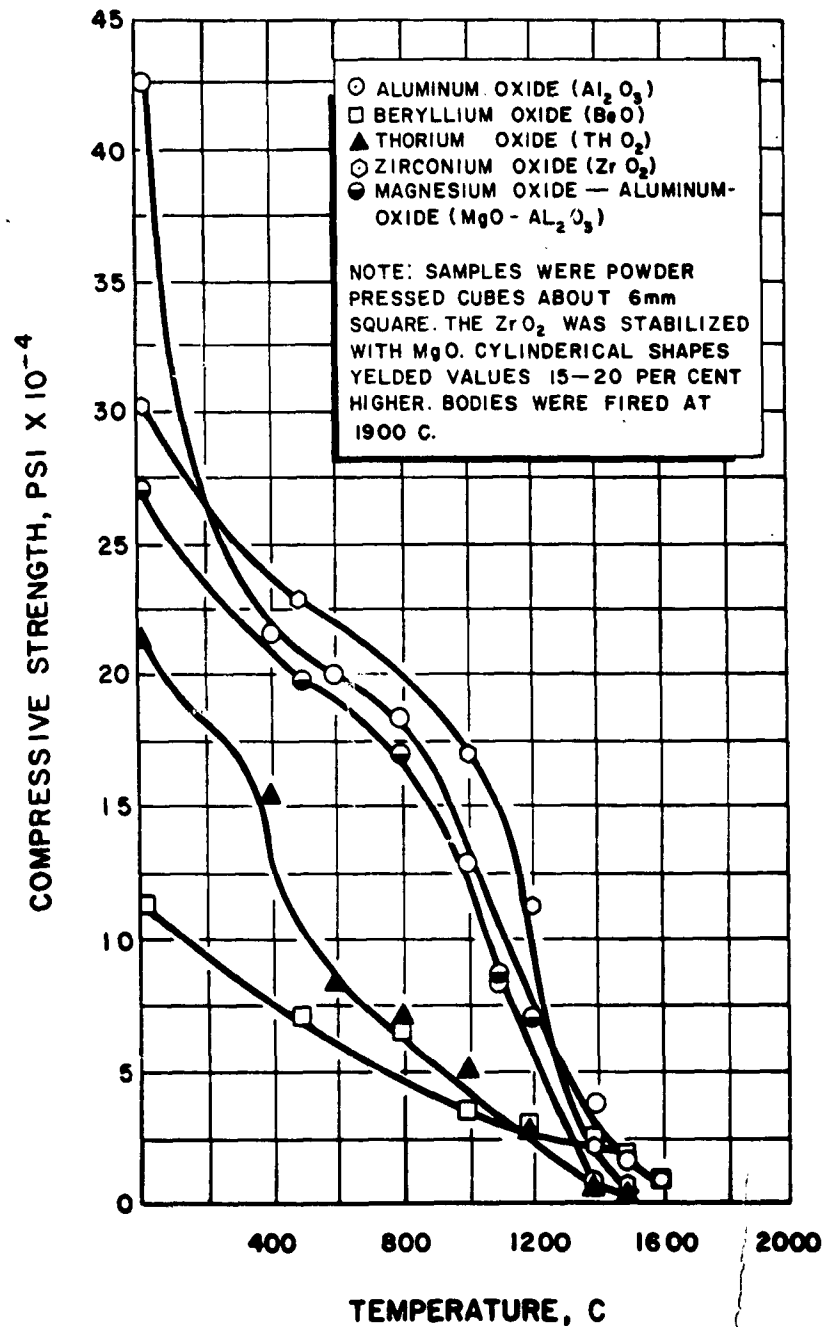


Figure 18. Effect of Temperature on the Compressive Strength of Some Refractory Oxides

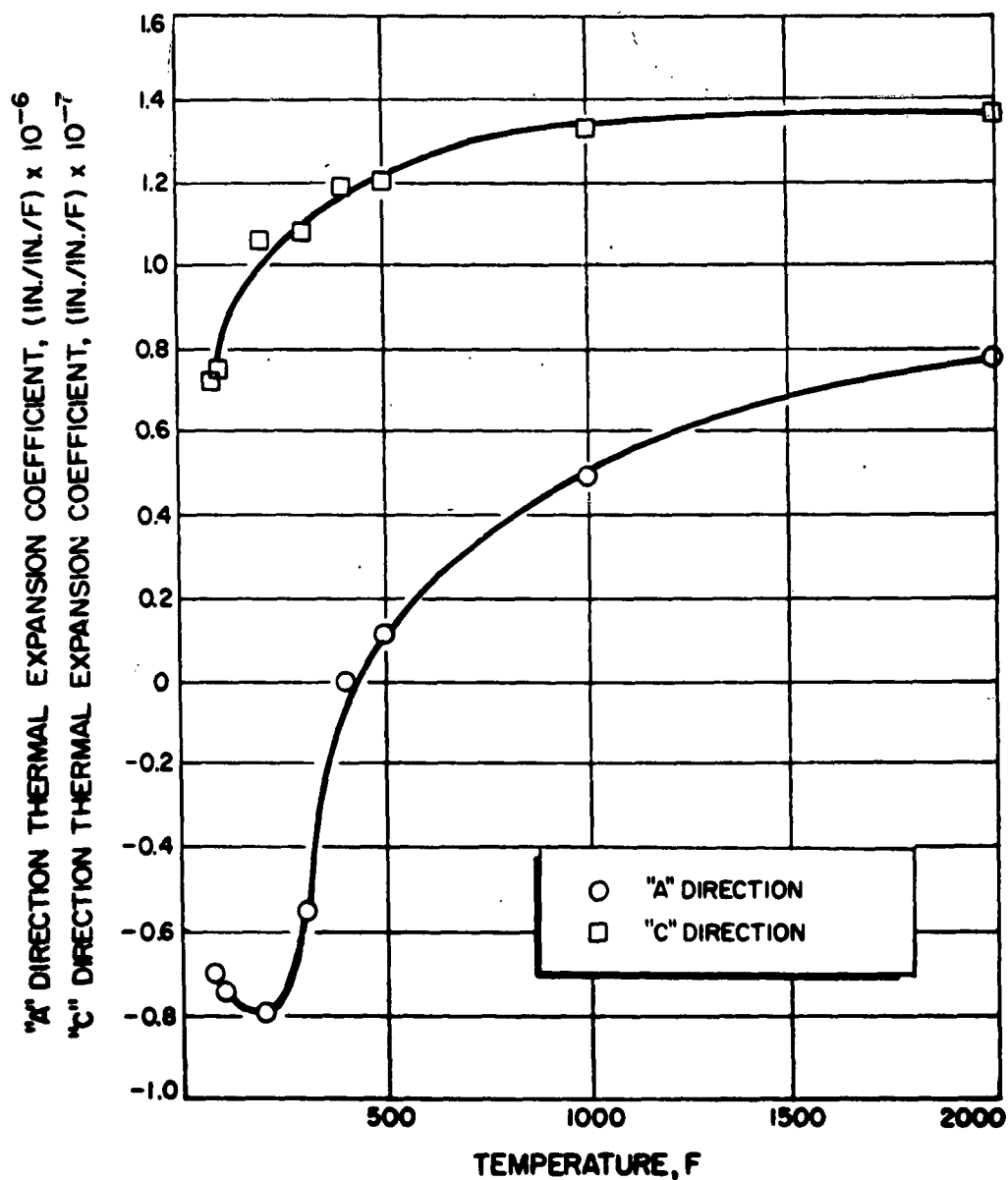


Figure 19. Effect of Temperature on the Thermal Expansion Coefficient of Pyrolytic Graphite

III-134

B-3390-3

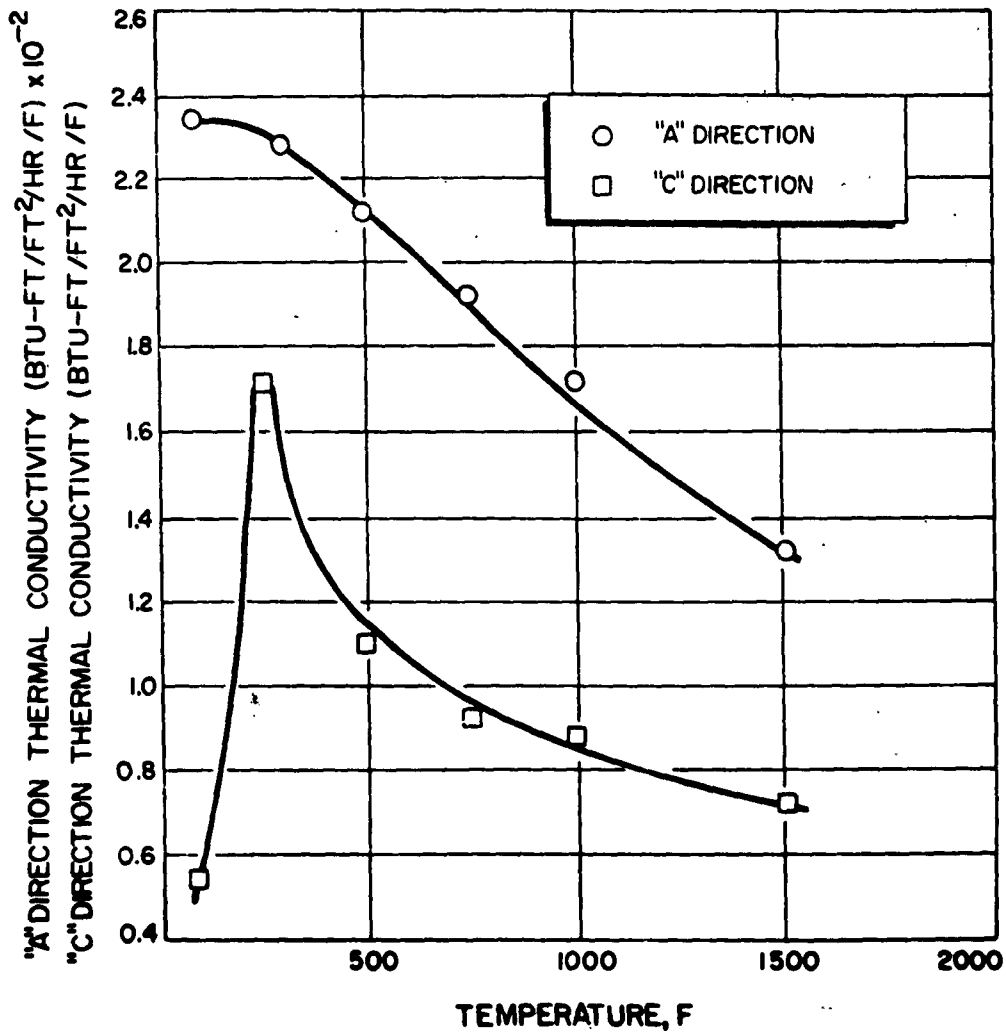


Figure 20. Effect of Temperature on the Thermal Conductivity of Pyrolytic Graphite

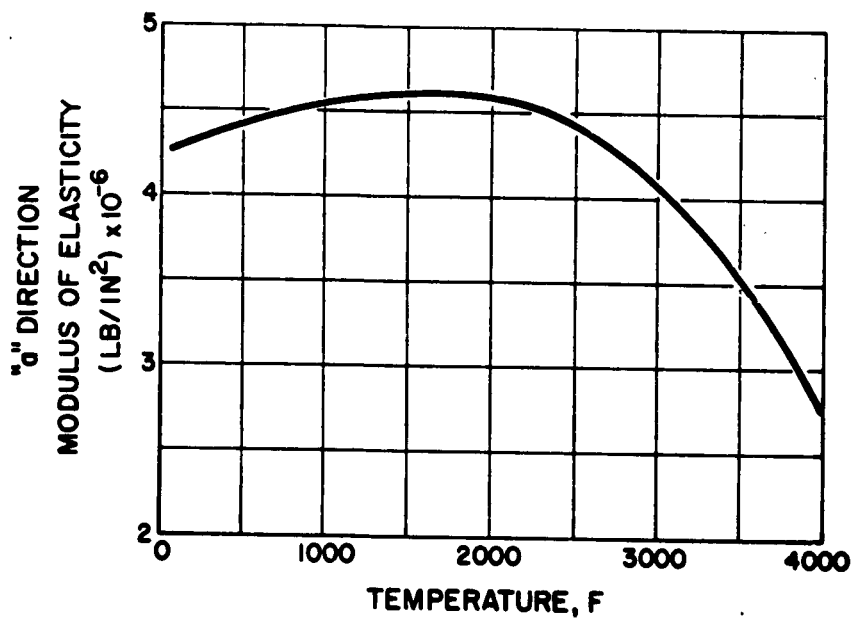


Figure 21. Effect of Temperature on the Modulus of Elasticity in Tension of Pyrolytic Graphite

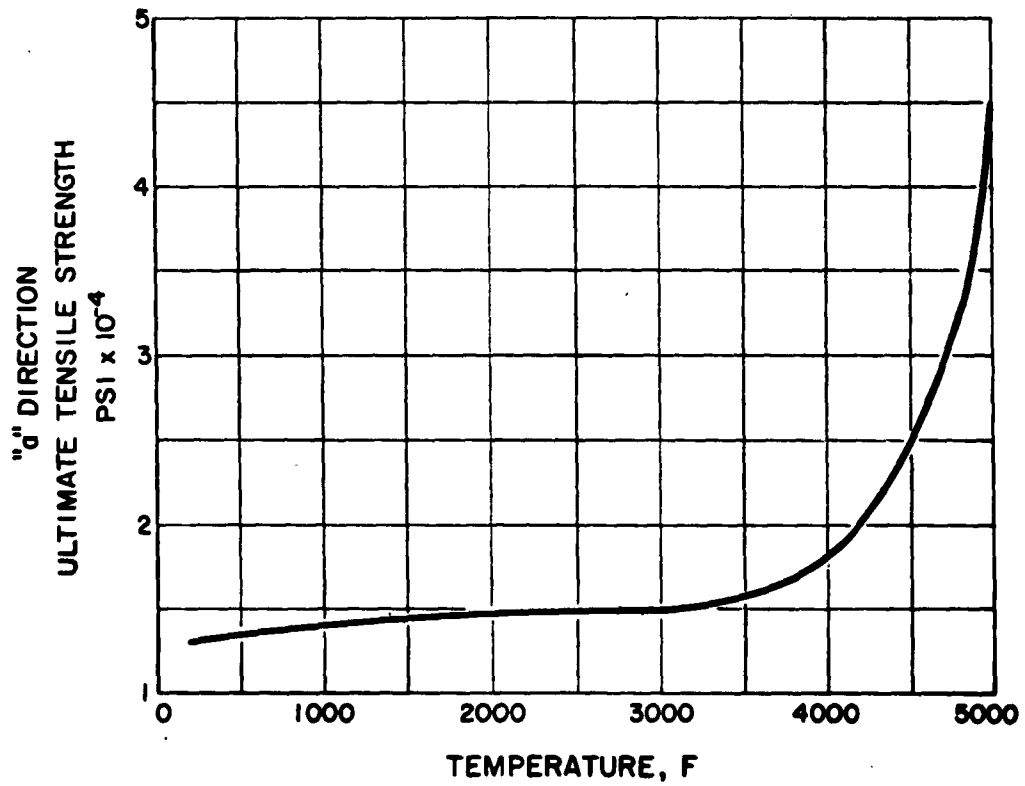


Figure 22. Effect of Temperature on the Ultimate Tensile Strength of Pyrolytic Graphite

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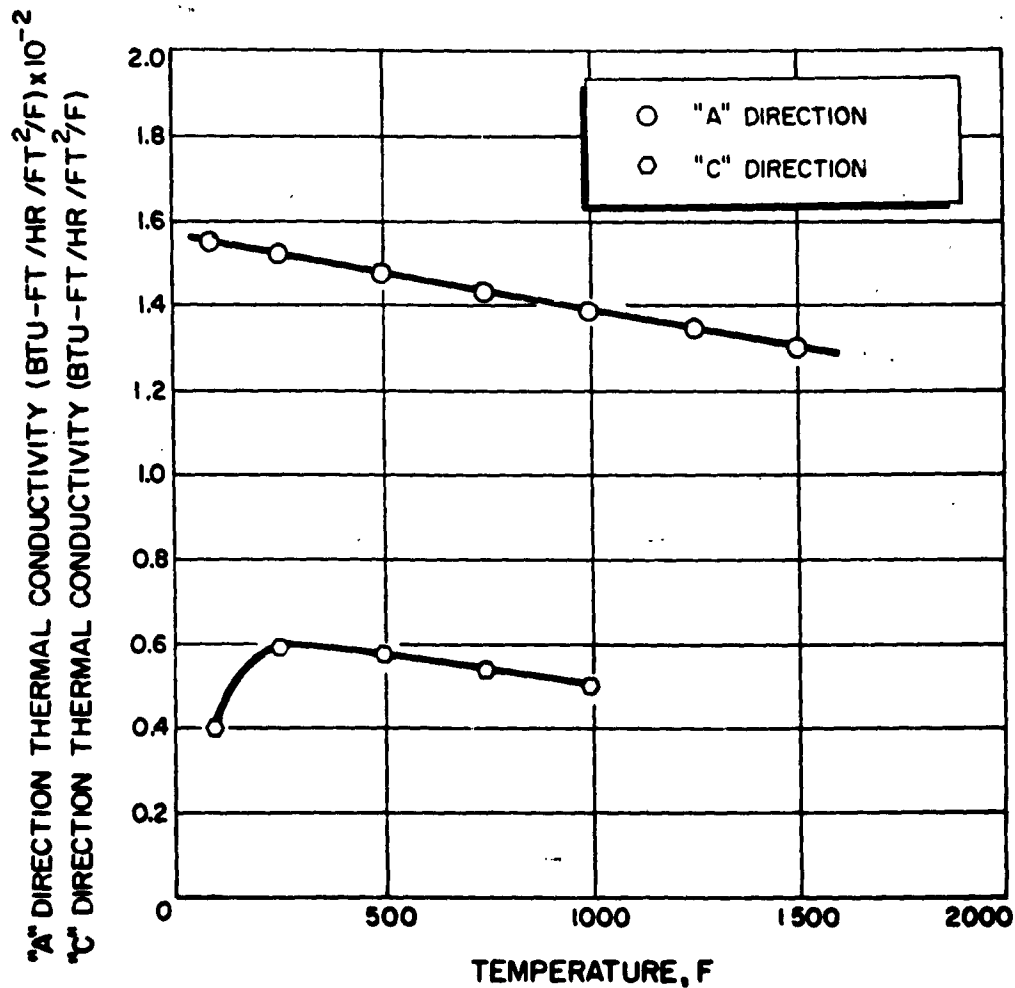


Figure 23. Effect of Temperature on the Thermal Conductivity of Boron-Pyrographite

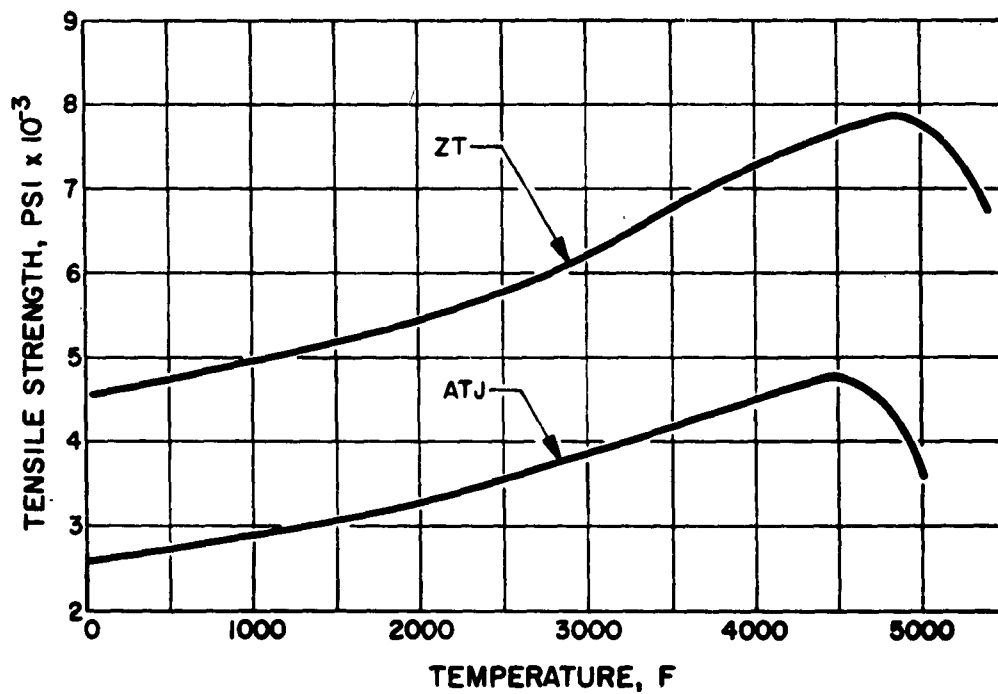


Figure 24. Tensile Strength of ZT and ATJ Graphite (Parallel to Grain) as a Function of Temperature

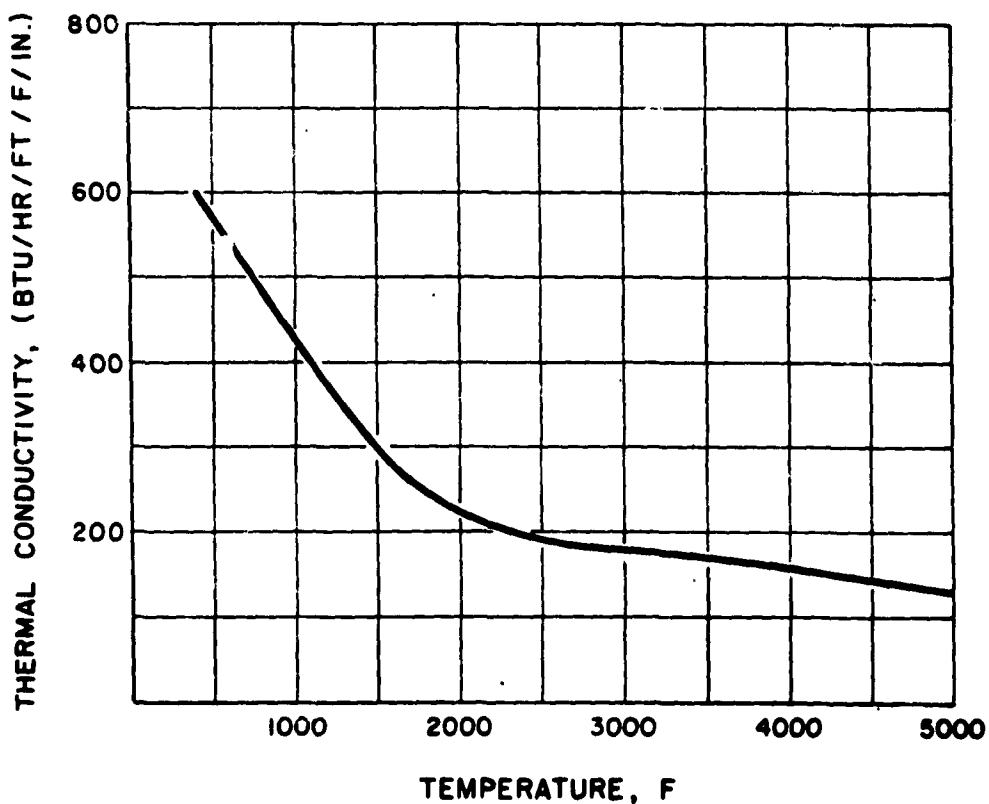


Figure 25. Thermal Conductivity of ATJ Graphite as a Function of Temperature

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B-3350-3

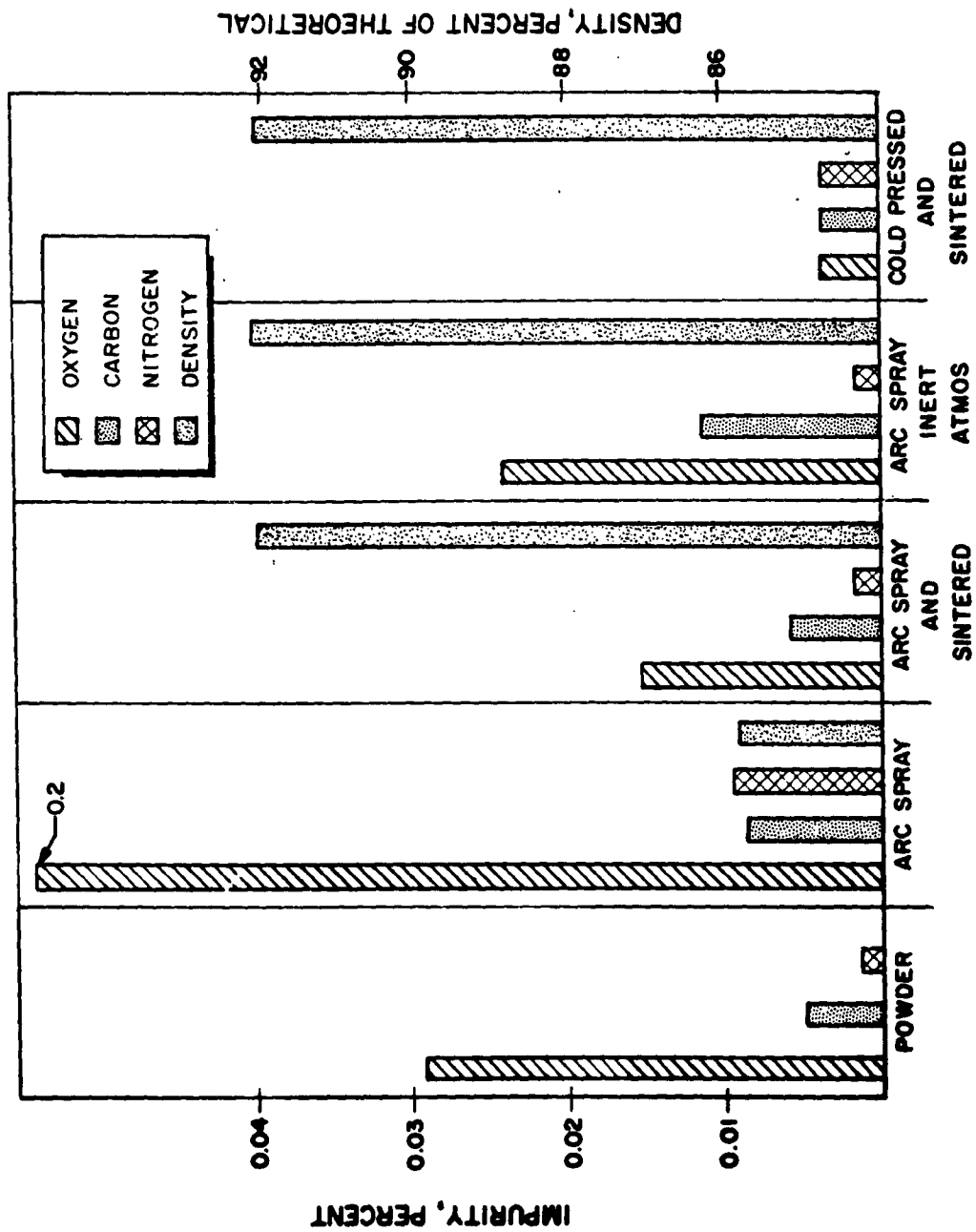


Figure 26. Typical Oxygen, Carbon, and Nitrogen Impurities Levels and Densities of Tungsten

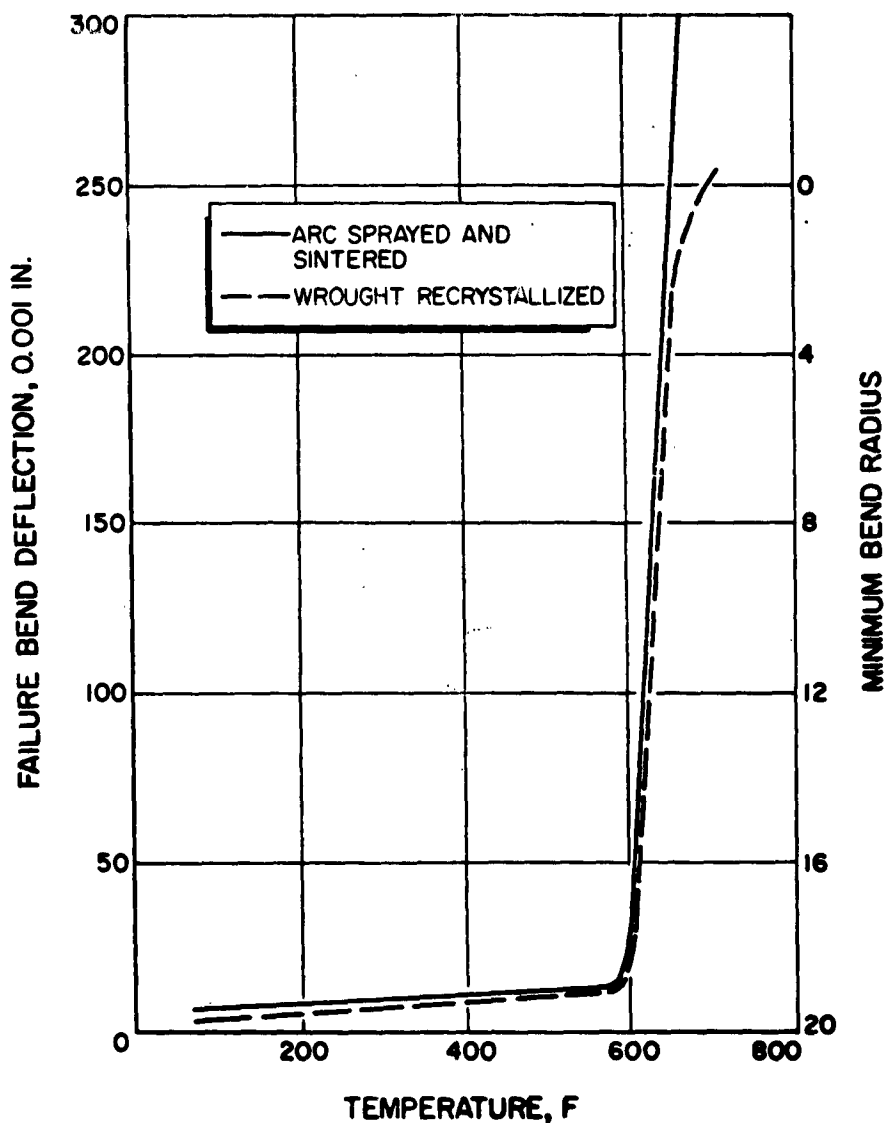


Figure 27. Effect of Temperature on the Bend Ductility of Recrystallized Tungsten

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B-3590-3

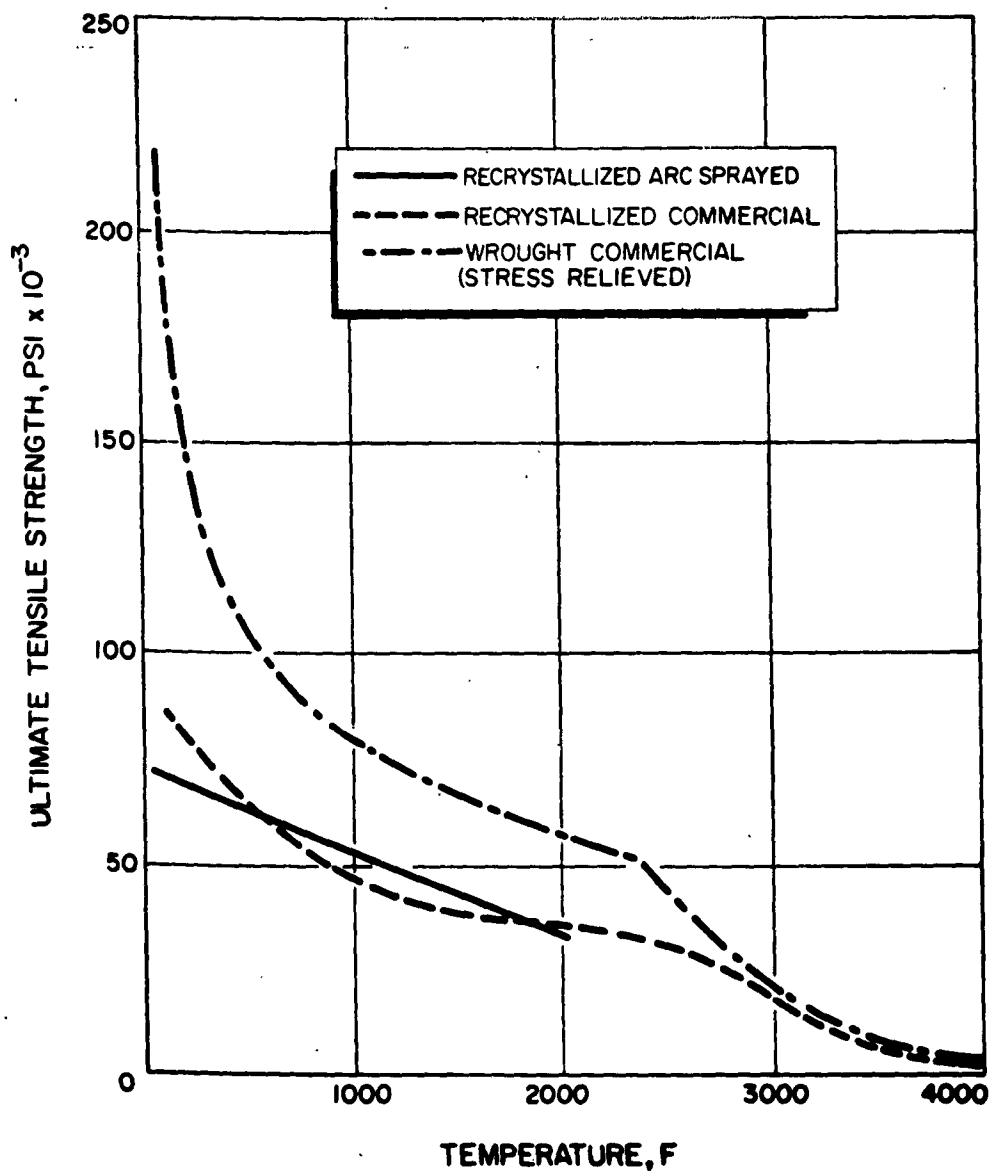


Figure 28. Tensile Properties of Tungsten